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Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

The Continuous Dyeing of Vat Dyes
J. Ardron, M. R. Fox, and R. W. Speke

The Reduction Properties of Vat Dyes
W. J. Marshall and R. H. Peters

A New Method for the Application of Oxidation Blacks
C. Zuber and B. Jomain

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The Reaction between Wool and Nickelammonium Hydroxide
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J. W. Bell and C. S. Whewell

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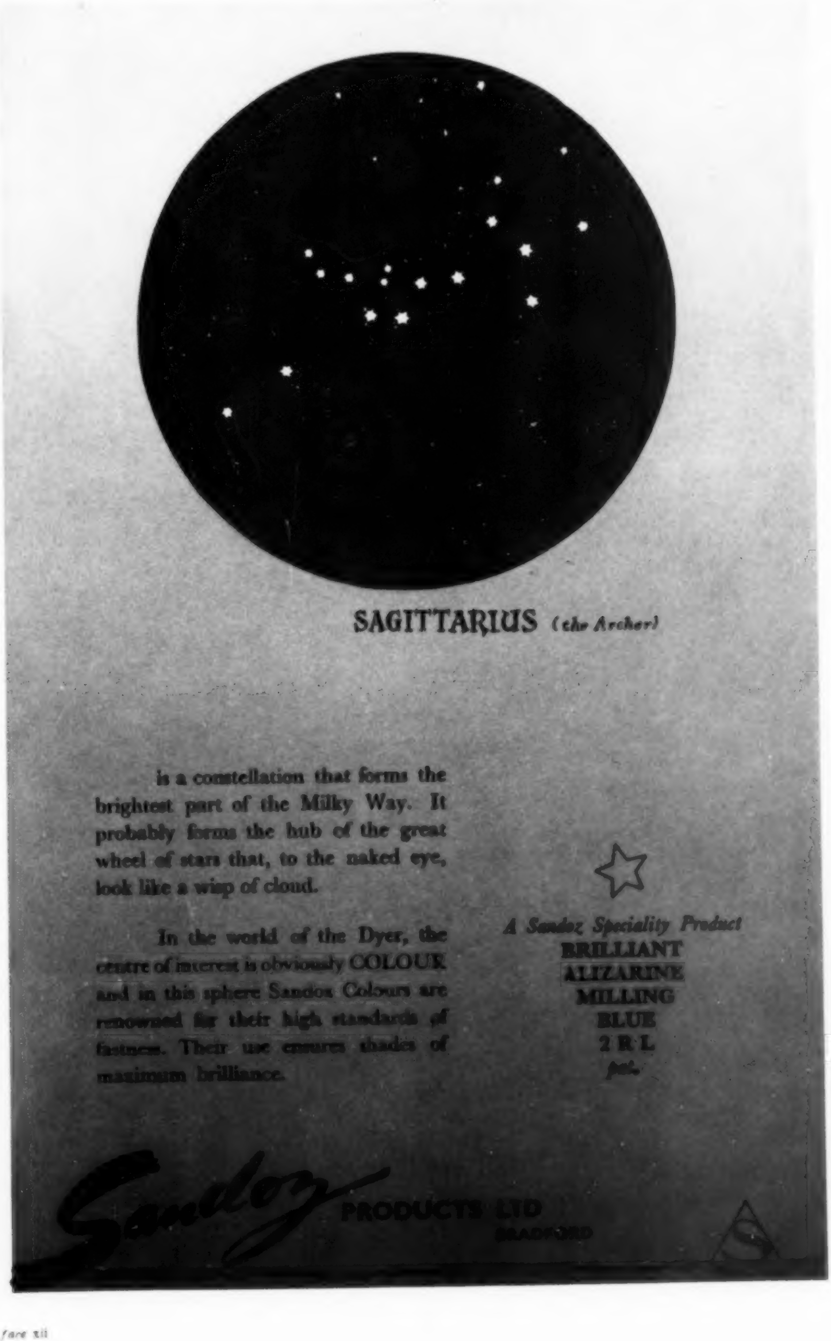
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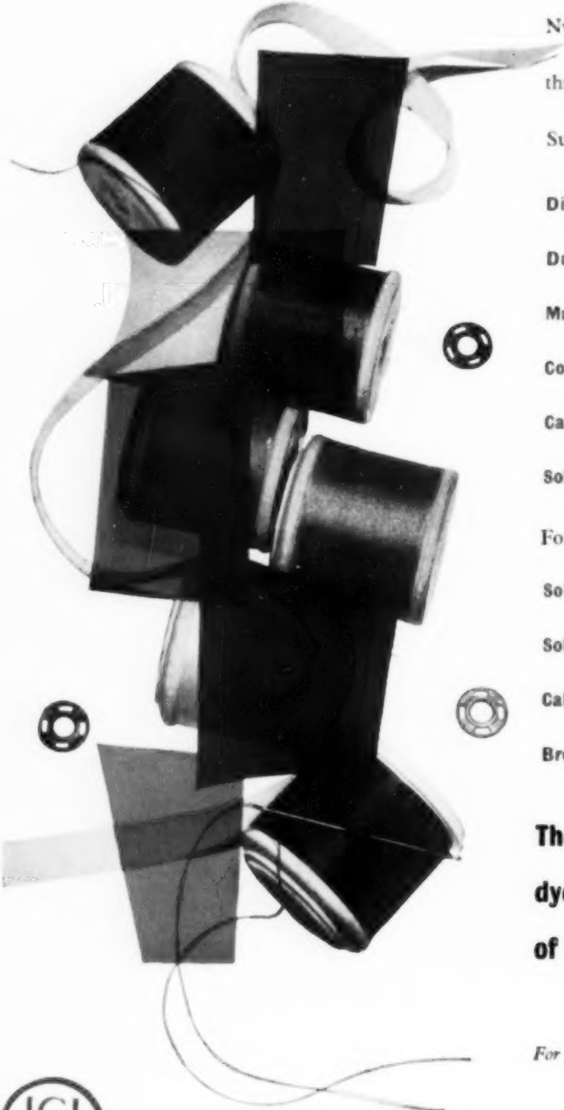

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THE JOURNAL
OF THE
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Volume 68—Number 6 JUNE 1952 *Issued Monthly*

PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE—V

Method of Test for Determining the Fastness to Daylight of Coloured Textiles

Second Draft Revision (11th March 1952) of *B.S.* 1006

LIGHT FASTNESS SUBCOMMITTEE

INTRODUCTORY NOTE

This draft revision of *B.S.* 1006 : Part 1 is reproduced here in order that members of the Society may offer any comments for consideration by the Fastness Tests Co-ordinating Committee before the document is submitted to the British Standards Institution in final form.

Comments should be forwarded to the General Secretary, Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorks., and should be received within one calendar month of publication in the Journal.

FOREWORD

Work on the production of standards for determining the fastness of coloured textile materials was initiated by the Society in 1927, and resulted in the selection of two parallel series of dyed standards of fastness to light, the description of which is given in the Society's *Report on the work of its Fastness Committee in fixing Standards for Fastness to Light, Perspiration and Washing*, published in 1934.

These two series of standards, a blue and a red, each comprised seven dyeings of increasing fastness, numbered 2–8, and graded in such a manner that the fastness to light of the series increased in geometrical progression; that is, so that each member of the series required double the exposure of the preceding standard to show the same degree of fading.

After the publication of the Society's Report, comments on the standards were invited, and close contact was established with the Fastness Committee of the American Association of Textile Chemists and Colorists and the Echtheitskommission of the Verein Deutscher Chemiker, which had each selected their own set of standards for fastness to light. Results were compared, and discussion was initiated with a view to the international adoption of a common range of standards. At the same time, certain of the standards were known to be imperfect, and the Society's Fastness Committee carried out further investigations with a view to improving them.

Although it was not found possible to reach complete agreement on all the standards with the above-named societies, considerable progress was made, and as a result a series of eight blue standards was issued. These improved standards were published in the *Journal of the Textile Institute* and in the Society's *Journal*[1] as Tentative Textile Standards.

In accordance with an agreement between the Textile Institute and the British Standards Institution, the standard was forwarded to the B.S.I. for development as a British Standard. It was approved by the Textile Divisional Council and was published, under the authority of the General Council of the B.S.I., as British Standard 1006 on 16th February 1942.

At a symposium on Photochemistry in relation to Textiles held by the Society in 1949[2] it was revealed that serious differences in assessment had resulted from different interpretations of the wording of this standard and also that the spacing of the standards was not entirely satisfactory.

This revised British Standard is based upon the recommendations of the Light Fastness Subcommittee and approved by the Society's Fastness Tests Co-ordinating Committee. It differs from the original standard in two respects, viz.—

(1) The extent to which the pattern must have changed before assessment is made has been clearly specified by reference to a series of fixed contrasts.

(2) To provide a fuller description of the light fastness of patterns which change to a small extent very rapidly but which are then quite resistant to prolonged exposure, provision has been made to record both these features, instead of only one as hitherto.

METHOD OF TEST

Object

1. The object of daylight fastness tests is to determine how a coloured pattern withstands the action of daylight in relation to certain standards.

This simple comparison may be complicated, however, by the fact that many patterns show a slight change after a relatively short exposure, but do not show further change if the exposure is prolonged. As an example, a pattern is exposed until a change can just be seen; examination of the standards exposed simultaneously reveals that Standard 2 has changed to the same extent. When, however, the exposure is prolonged until a further change in the pattern can be observed, Standard 2 may become almost white and the standard which now has changed to the same extent as the pattern is Standard 5. As both the slight initial change and the ability of the pattern to withstand prolonged exposure are important, it is clear that the light fastness of such patterns can be adequately described only by giving figures for both effects. The method of exposure and assessment takes account of this behaviour.

Note—The method described is intended to be used for coloured materials; when colouring matters (e.g. dyes) are being tested, it is recommended that this method is used but, as the light fastness of these products depends on the depth of dyeing, this should also be stated.

British Standard Patterns

2. British Standards of Fastness to Light shall consist of eight blue dyeings and shall be dyed on wool material. The dyes used shall be as follows—

1. Brilliant Wool Blue FFR Extra (IG)
2. Brilliant Wool Blue FFB Extra (IG)
3. Brilliant Indocyanine 6B (IG)
4. Polar Blue G Conc. (Gy)
5. Solway Blue RS (ICI)
6. Alizarine Light Blue 4GL (S)
7. Soledon Blue 4BCS Powder (ICI)
8. Indigosol Blue AGG (DH)

Note—It has been found that, when repeat preparations of the dyed standards are made, the amount of dye required to match the previous pattern is often different from that originally used; as strength references can therefore be misleading, they will no longer be given. The 1952 standards are identical with those of 1940 with the exception of Standard 2, which has been increased in depth of dyeing in order to improve the spacing.

The British Standards Institution has agreed that the standard patterns issued by them shall be prepared and approved by the Society of Dyers and Colourists. Sets of standards can be obtained from the British Standards Institution; single standards will also be supplied on request.

METHOD OF USE

3. The material under test and the standards which are exposed simultaneously shall be partly covered with an opaque card (see Appendix A) and exposed to daylight. They shall be protected from the weather by exposing under window glass facing due South and sloping at an angle of 45°, care being taken to ensure that obstructions cannot cast shadows on the patterns; adequate ventilation shall be provided.

Note—The use of sources other than daylight (e.g. fading lamps) is common practice, and the results obtained under such conditions are undoubtedly valuable for rapid assessment. They differ in many cases, however, from the results obtained by daylight exposure. When artificial light sources are used, therefore, it is recommended that exposure and assessment be carried out as for daylight. It must be clearly stated, however, that the results have been obtained from exposures to artificial light sources and therefore are not in accordance with the specification.

The pattern under test shall be frequently inspected by removing the opaque card, and when a change can just be perceived, the number of the standard showing a similar change shall be noted.

Note—If a pattern is phototropic, it shall be allowed to condition in the dark at room temperature for 2 hr. before any assessment is made.

The exposure shall then be continued until the contrast between the exposed and the covered portions is equal to the contrast between the two grey patterns illustrating Grade 3 on the Geometric Grey Scale (see Appendix C). Part of the exposed pattern and the standards shall then be covered, and exposure continued until the contrast between the fully exposed and the unexposed portions is equal to Grade 2 on this grey scale.

When a pattern has a light fastness of 7 or over, it would require unduly long exposure to produce a contrast equal to Grade 2; moreover, this contrast would be impossible to obtain when the light fastness is 8. Assessments in the region 7 to 8 shall therefore be made when the contrast produced on standard 7 is equal to Grade 3, the time required to produce this contrast being long enough to eliminate any error which might result from inadequate exposure.

METHOD OF ASSESSMENT

4. The pattern now shows three separate regions—one unexposed and two which have changed to different degrees; these changes shall then be compared with the changes which have occurred in the standards. The light fastness of the pattern shall be the number of the standard which shows similar changes. If the pattern shows an effect between two standards, then the appropriate half-grade shall be given expressed in the form e.g. 3–4. Any pattern more fugitive than Standard 1 shall be graded 1.

If the light fastness of a pattern assessed by this method is 4 or higher, then the preliminary assessment based on the first perceptible change becomes significant, and if this preliminary assessment is 3 or lower, then it shall be included in any statement relating to the light fastness of the pattern; this preliminary assessment shall be distinguished by enclosing it in brackets.

For example, an assessment of 6 (3) indicates that the pattern changes very slightly at the stage when Standard 3 just begins to fade; apart from this, however, the resistance of the pattern to exposure is equal to that of Standard 6.

Note—It must be understood that the term "changes in the pattern" includes not only true fading, i.e. destruction of colour, but also changes in hue; this is of particular importance with regard to the initial change. When the change in hue is appreciable, it is recommended that it be recorded; e.g. a yellow pattern which becomes orange on exposure should be assessed "3, redder", the number 3

indicating that on exposure the changes in the pattern (yellow becoming redder) appear visually equal to the changes in Standard 3 (blue becoming paler).

APPENDIX A

The object of partly covering the pattern under test and the standards is to produce by exposure two areas (which have changed to different extents) each adjacent to an unexposed region. Any method of covering which achieves this object can be used, and Fig. 1 illustrates one simple method.

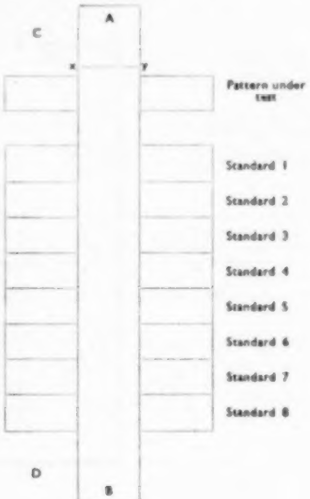

Fig. 1

The top of the thin opaque card *AB* (hinged at *xy*) is securely fastened to the board on which the patterns are mounted; the portion of the card which covers the patterns is then firmly held down to prevent buckling. The free portion of the card is periodically lifted during exposure, so that the pattern under test can be inspected. After inspection, the card is replaced exactly in its original position. When a change in the pattern can just be perceived, the number of the standard showing a similar change is noted, and exposure continued. When the contrast produced on the pattern is equal to that illustrating Grade 3 on the Geometric Grey Scale, another opaque card *CD* is fixed overlapping the original card, and exposure continued until the contrast between the fully exposed and the unexposed portion of the pattern is equal to Grade 2; both opaque cards are then removed, and the pattern is assessed.

APPENDIX B

It is realised that the method previously described requires one set of standards for each pattern under test, and that this method would be impracticable when a large number of patterns have to be tested concurrently. The following method is therefore recommended as a compromise which enables a number of patterns differing in light fastness to be assessed against a single set of exposed standards with negligible loss of accuracy.

The patterns and standards are mounted as shown in Fig. 2.

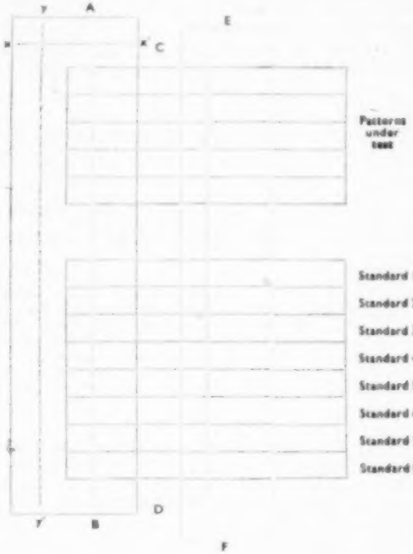

Fig. 2

The opaque card *AB*, hinged at *xx'* or *yy'*, is placed at the edge of the patterns and standards covering one-quarter of the total length of the strips.

This card is periodically removed during exposure, and the *standards* are inspected; when a change in Standard 3 can just be perceived, the patterns are inspected, and any change that has occurred is assessed by comparison with the changes that have occurred in Standards 1, 2, and 3. The card *AB* is then replaced in exactly the same position. This preliminary assessment shall be recorded when the final assessment is 4 or higher, as previously described.

Exposure is continued until a change in Standard 4 can just be perceived; at this point a further cover *CD* is fixed in the position shown, overlapping the first cover.

Exposure is continued until a change in Standard 6 can just be perceived; the final cover *EF* is then fixed as shown, the other two cards remaining in position. Exposure is then resumed until either—

(*a*) A contrast is produced on Standard 7 equal to the contrast illustrating Grade 3 on the Geometric Grey Scale or

(*b*) A contrast equal to Grade 2 has been produced on the most resistant pattern

whichever occurs first. The three opaque cards are then removed, and the patterns assessed by comparison with the standards.

APPENDIX C

The Geometric Grey Scale will shortly be available from the Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorks. It consists of five pairs of grey patterns showing a degree of contrast which increases geometrically. Until this scale is available, however, an indication of the extent to which exposure must be carried before patterns are assessed can be obtained by reference to Grey Scale No. 2 of the Society of Dyers and Colourists: Grade 2 on the Geometric Grey Scale is approximately equal to Grade 4 on the Grey Scale No. 2; Grade 3 on the Geometric Grey Scale is about one-half of this contrast.

(*Received 27th March 1952*)

References

[1] J.S.D.C., **56**, 273 (June 1940).
[2] *Ibid.*, **65**, 585 *et seq.* (Dec. 1949).

PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE—VI

The Rates of Fading of the S.D.C. Light Fastness Standards (*B.S.* 1006)

R. H. Ricketts

The rates of fading of the S.D.C. light fastness standards 1 to 6 have been determined by exposing portions of each standard for different times in a fading lamp and finding the difference between the exposed and unexposed patterns, optically and visually. The reflectance curves of the patterns have been obtained, and two colour difference formulæ used to calculate the colour change, on exposure, in just perceptible steps. It is shown that the results obtained optically do not correspond to the observed differences. In view of this, the differences have been estimated visually against a grey scale, and the results obtained show that the spacing of the standards 1 to 6 is satisfactory.

INTRODUCTION

The S.D.C. light fastness standards consist of a series of eight blue dyeings on wool. These were selected, after an extensive examination of potentially suitable patterns, so that each standard was twice as fast as the next lower standard (i.e. would require twice the exposure to produce the same degree of change).

Recently, the spacing of these standards has been criticised, particularly in America. Thus Godlove [1] exposed each standard so as to produce a small degree of fading, each standard receiving twice the exposure of the next lower standard. He showed that the colour differences between exposed and unexposed standards, when calculated from spectrophotometric reflectance curves, were not equal.

In a reply [2] to this paper, the F.T.C.C. pointed out that the degree of fading was much less than was intended when the standards were established. The Committee also stated that they had had no experience of the relationship between the quantity derived from the Adams formula and the visual judgment of fading, and they were not satisfied that this formula was suitable.

In view of Godlove's criticism and the Committee's reply, it was felt desirable to determine the rate of fading of each standard, using optical and visual methods of measurement, and to compare the spacing of these standards at different degrees of fading.

EXPERIMENTAL

Pieces of the S.D.C. standards, 2 in. × 1½ in., were exposed in a modified Kelvin, Bottomley & Baird fading lamp. Owing to fluctuations in voltage, variations in the carbons, and other factors, exposing for definite lengths of time is not a satisfactory method of measuring the exposure. The fading lamp was therefore fitted with a light flux-time integrator of the type described by Atherton and Seltzer [3]. To avoid errors due to variations in illumination round the arc, only the spaces adjacent to the photocell were used. The reflectance curves of the exposed and unexposed patterns were measured on the General Electric recording spectrophotometer. By integrating these curves for Illuminant C, using thirty selected ordinates, the C.I.E. tristimulus values X, Y, Z of each pattern were obtained [4].

Adams Formula

Several formulæ for expressing small colour differences as a single number have been reported in the literature, and one of the simplest to apply, based on the Adams colour space, has been described by Nickerson [5].

The X, Y, Z equivalents of the Munsell value scale V_x, V_y, V_z are obtained from tables, and the colour difference ΔE between two patterns is obtained from the formula—

$$\Delta E = f\{[\Delta(V_x - V_y)]^2 + [0{\cdot}23\ \Delta V_y]^2 + [0{\cdot}4\ \Delta(V_z - V_y)]^2\}^{\frac{1}{2}}$$

when $f = 1$ the difference is given in Adams units and when $f = 40$ the difference is given in "just perceptible steps" or N.B.S. (National Bureau of Standards) units.

The differences, in N.B.S. units, between exposed and unexposed pieces of the standards have been calculated by this method.

In Fig. 1 the colour differences ΔE are plotted against light integrator units for each of the standards. It can be seen that by this method standards 1, 2, and 3 appear very close together, and standard 4 appears faster than standard 5. Since this is contrary to observed results, either the method of exposure is not reproducible or the

method of calculation is at fault. To check the former, further pieces of standards 3 and 4 were exposed for approx. 600 and 1000 units, their reflectance curves measured, and the colour differences calculated as before. These differences are given in Table I; in column 10 the colour differences corresponding to the exposures given have been read from the graph (Fig. 1), and in the last column the percentage error of the repeat determination is given. As the average error is less

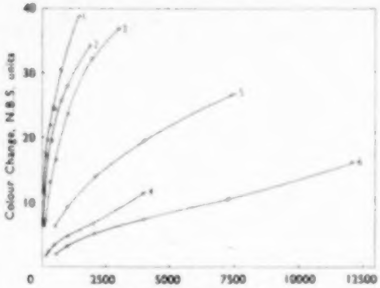

Fig. 1

than 5%, it can be concluded that the anomalous results are not due to the method of exposure. It would appear, therefore, that the Adams formula is not suitable for expressing the degree of fading of these standards.

Table I

Standard	Exposure (integrator units)	X	Y	Z	V_x	V_y	V_z	ΔE (N.B.S. units)	ΔE (from Fig. 1)	Error (%)
3	0	8·68	5·92	34·38	3·47	2·85	5·92	0	—	—
	605	8·49	7·03	26·37	3·44	3·11	5·27	18·4	18·0	+ 2%
	1126	8·98	8·10	24·64	3·53	3·33	5·12	26·8	25·2	+ 6%
4	0	7·0	7·13	20·92	3·15	3·13	4·76	0	—	—
	605	7·70	7·77	19·89	3·28	3·26	4·66	3·9	3·9	0
	1126	8·16	8·17	20·27	3·37	3·34	4·70	4·8	5·0	− 4%

Godlove Formula

An "improved colour-difference formula" described recently by Godlove[6] has been used to calculate the colour changes of the exposed and unexposed standards. This formula gives the colour change I_4 as follows—

$$I_4 = [2C_1C_2\,H + (\Delta C)^2 + (4\Delta V)^2]^{\frac{1}{2}}$$

where C_1 and C_2 are the respective chromas of the two patterns, ΔC is the change in chroma, ΔV the change in value, and $\phi H = 1 - \cos 3{\cdot}6^\circ\,\Delta H$, ΔH being the change in hue. Tables are provided for obtaining ϕH from ΔH. The value, hue, and chroma are Munsell renotations, and these were obtained from the Newhall-Nickerson-Judd[7] graphs for converting C.I.E. co-ordinates to Munsell renotations.

The unit of colour difference in the I_4 formula is one chroma step, compared with the unit used in the Nickerson index of fading, which is $\frac{1}{5}$ chroma step. The Adams unit is [illegible] of the Nickerson unit; and as, in the formula used earlier, the units used were 40 times the Adams unit, the colour differences obtained from the Godlove formula have been multiplied by [illegible] to give the same magnitude as the N.B.S. unit.

The values of I_4 in N.B.S. units have been plotted against the exposure in Fig. 2. Although some improvement in spacing is obtained, standard

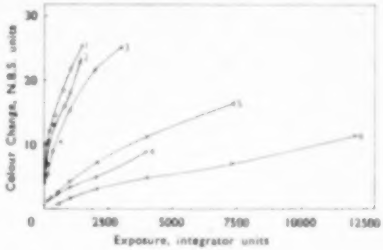

Fig. 2

5 still appears to be more fugitive than standard 4, and it would appear, therefore, that the Godlove formula also is not suitable for expressing the colour changes of these faded standards.

Visual Assessment

In view of the anomalous results obtained optically and because these standards are used for the visual assessment of light fastness, it was decided to assess the patterns visually against a grey scale. The latter was prepared by dyeing botany serge with Alizarine Light Grey G (S) in a range of depths and measuring the reflectance curves of the patterns on the G.E.C. recording spectrophotometer. As these patterns approximate to neutral greys, they differ only in Munsell value, and it is therefore necessary to measure only the Y value. The difference between patterns can be calculated in N.B.S. units using the Adams formula. For neutral greys $V_x - V_y$ and $V_z - V_y$ are equal to zero, and the formula reduces to—

$$\Delta E = 0{\cdot}23 f \Delta V_y$$

From the results, a series of patterns showing 3, 5, 8, 12, and 18 just perceptible steps were obtained. The measurements are given in Table II.

Table II

Pattern	Y	V_y	ΔV_y	ΔE (N.B.S. units)
A	9·73	3·63	0	0
B	11·66	3·95	0·32	2·9
C	13·17	4·17	0·54	5·0
D	15·83	4·53	0·9	8·3
E	19·08	4·94	1·31	12·0
F	25·08	5·55	1·92	17·6

The faded specimens were prepared for assessment in the following manner—one piece, 1 in. × 1½ in., of each exposed pattern and a piece of the unexposed standard of equal size were mounted side by side on a card 2 in. × 1½ in. A grey scale was prepared in a similar fashion, a piece of each of the patterns B, C, D, E, and F being mounted adjacent to pieces of pattern A.

The patterns were assessed against a matt black background illuminated by North daylight. The

patterns were compared with the pairs of the grey scale, the object being to compare the contrast between exposed and unexposed patterns with the contrast between the various pairs of the grey scale. Where the degree of contrast between patterns was found to be intermediate between the contrast shown on the grey scale an estimated figure was given. The patterns were assessed by ten operators.

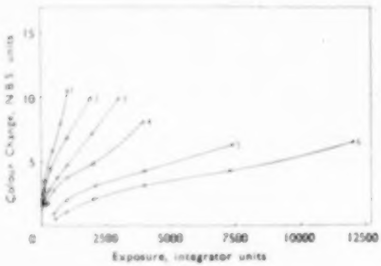

FIG. 3

The average assessment has been plotted against the recorded light units in Fig. 3. The spacing of the standards on this graph is more uniform, and they are in the correct order. If these standards are spaced so that each is twice as fast as the preceding standard, then the logarithm of the amount of light required to produce the same degree of fading on each standard, plotted against the number of the standard, should give a straight line of slope $\log_{10}2$.

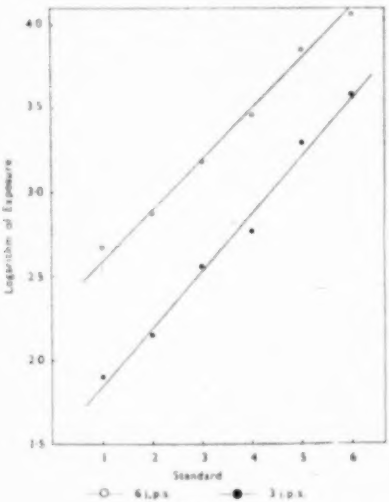

FIG. 4

In Fig. 4 this has been carried out for 3 and 6 just perceptible steps (j.p.s.), these being the degrees of fading recommended in the revised draft of *B.S.* 1006: Part 1.

Examination of the graph shows that at a fade corresponding to 6 j.p.s. the standards lie close to a line of slope $\log_{10}2$. At 3 j.p.s. the standards lie close to a straight line of slope $\log_{10}2{\cdot}2$.

The results obtained by the three methods described above are compared in Table III.

TABLE III

Standard	Exposure (integrator units)	Colour Difference (N.B.S. units) Adams	Godlove	Visual
1	46	11·7	6·4	2·2
	114	17·6	10·3	3·5
	426	24·8	14·7	5·9
	740	30·5	18·5	7·9
	1029	34·1	21·8	10·5
2	54	9·4	5·7	1·9
	102	11·0	7·0	2·7
	361	19·5	13·1	4·4
	1005	27·9	18·0	6·8
	1884	34·2	22·9	9·9
3	77	6·4	5·0	1·8
	293	13·1	9·0	2·8
	625	16·4	10·5	3·7
	1004	23·6	15·4	4·7
	1961	32·2	21·6	7·1
	3010	36·8	25·1	9·8
4	148	1·8	1·4	1·6
	247	2·4	1·9	1·7
	483	3·5	2·3	2·9
	1004	4·8	3·6	3·8
	2002	6·7	5·0	4·9
	3950	11·4	8·9	8·0
5	498	6·3	2·7	0·9
	976	9·2	4·4	2·0
	2083	14·0	7·2	3·1
	3982	19·4	11·2	4·2
	7361	26·5	16·4	6·2
6	535	2·0	0·8	0·6
	976	3·3	1·7	1·1
	2082	5·1	3·2	2·1
	3982	7·2	4·9	3·1
	7271	10·5	7·0	4·2
	12070	16·1	11·3	6·4

Comparison of Three Methods

Examination of Table III shows that, when the colour change on exposure of the S.D.C. standards is calculated from spectrophotometric data, the results differ from the observed change assessed visually against a grey scale, the results obtained by calculation being greater. The differences between the calculated and observed results are greatest for the standards 1, 2, 3, and 5. These are very bright patterns, and in the early stages of exposure they become duller without much loss in depth. For standard 4, which is the dullest of the six standards, the observed and calculated results are of the same order.

CONCLUSIONS

The specification for light fastness assessment requires that a pattern under test be exposed for

two periods so as to produce contrasts visually equal to the two specified contrasts of the Geometric Grey Scale. The results obtained by visual assessment show that the S.D.C. standards 1 to 6 are correctly spaced— i.e. each standard is twice as fast as the next lower standard— when exposed in accordance with this specification.

It is also shown that the two colour-difference formulæ used are not suitable for calculating the change on exposure of these standards. Criticisms of the S.D.C. standards based on these formulæ are therefore not valid.

* * *

The author wishes to acknowledge the assistance of Dr. I. M. S. Walls in the preparation and interpretation of the spectrophotometric data.

Dyehouse Laboratories
Imperial Chemical Industries Ltd.
Hexagon House
Manchester 9

(Received 24th April 1952)

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PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE—VII

The Spacing of Scales for Assessing the Effect on the Pattern in Fastness Testing

K. McLaren

The spacing of the first grey scales considered for assessing loss in depth in fastness testing has been found to be unsatisfactory, as the resulting assessments do not reveal the small but important differences between patterns of good fastness; this defect is inherent in all equally spaced scales. It is shown that satisfactory results are obtained when the scales are spaced geometrically. Four geometric grey scales have been prepared and sent on behalf of the International Standards Organisation to the Fastness Tests Committees of France, Germany, Switzerland, and the U.S.A. in the hope that agreement on the spacing may be achieved.

Perhaps the most important advance in fastness testing during recent years has been the greater precision in assessing the results of the tests. This has been achieved by comparing the tested patterns with "fixed standards", i.e. patterns which have been separately prepared (by dyeing or printing) to illustrate five grades of fastness. This principle, which is a logical development from the numerous standard "types" of the pre-war German tests, was introduced independently in Great Britain, Switzerland, and America.

In view of the universal desire for agreement, it is very pleasing to find that the British, Continental, and American fixed standards for staining assessment are practically equivalent.

Fixed standards for assessing the effect on the pattern, however, have not yet been adopted officially either in Britain or in America. The Fastness Tests Co-ordinating Committee considered this question very fully, and examined the suitability for this purpose both of the sixteen-step grey scale of the French–German–Swiss Fastness Committees and of the S.D.C. Grey Scale No. 2. The Committee concluded that neither scale was entirely satisfactory, for the following reasons.

When a coloured pattern is subjected to a fastness test the most common effect is that the pattern loses depth. This will vary between no visible effect (Grade 5) and very severe loss in depth (Grade 1). The simplest grey scale for such assessments will be equally spaced visually, and this was the method originally proposed by J.-P. Niederhauser and adopted by the French-German-Swiss Fastness Committees: in practice the scale was more complicated, because it covered patterns of different depths on a "sliding-scale" principle.

This equally spaced scale, however, suffers from a serious defect. The magnitude of the spacing is governed by the need to make Grade 1 representative of a severe change: Grade 4 is, therefore, one-quarter of this, and the undefined but permissible half-grade, 4–5, one-eighth. The Committee thinks that the spacing in the 3 to 5 region is too wide for the following reasons.

When a pattern merely loses depth in fastness testing, this can be expressed quantitatively as a percentage loss in the amount of dye present. It is generally accepted that, for patterns of average depth, a difference in strength of 5% can be perceived, even in the case of yellows, which are the worst shades for perceiving strength variations. In other words, if two patterns are dyed to complete exhaustion with 1·00% and 0·95% dye respectively, the stronger can be correctly chosen every time; in the case of shades such as browns, differences of 2·5% can usually be detected.

The highest rating of a fastness test is Grade 5, which is given when the pattern suffers no significant change. The next highest rating is 4–5, and this should be given only to patterns suffering approx. 5% loss in depth; on the French–German–Swiss scale, however, Grade 4–5 includes patterns suffering as much as a 15% loss in depth. Because it is broadly spaced, therefore, this scale is incapable of differentiating—

(*a*) two patterns, one of which is unchanged and the other suffers a 5% loss in depth— both would be rated 5

(*b*) patterns suffering 5–15% loss in depth— all would be rated 4–5

(*c*) patterns suffering 15–25% loss in depth— all would be rated 4.

This insensitivity in the region of good fastness is serious from the viewpoint of the dye user and the textile manufacturer, because it fails to separate patterns of excellent fastness from those which are very good, or to separate those which are very good from those which are merely of good fastness. This can be achieved only by making the spacing in the important 3 to 5 region much closer.

The Society intended the verbal description of Grade 3—"moderately changed"—to indicate the borderline between suitability and unsuitability, and the test controls for five of the wool fastness tests have been selected on this basis.

Before the advent of fixed standards, the only indication of the magnitude of the spacing of the five grades has been the accompanying subjective terms, and although different people will attach different meanings to the same word—and if differences in language are involved, the disagreement may be aggravated—the terms used to describe a Grade 3 fastness in other systems do invariably imply only a small change, viz.—

D.E.K.* (1939)	... Shade or depth of dyeing slightly altered (*etwas verändert*)
A.A.T.C.C. (1949)	... Good (fastness)

The Grade 3 change illustrated on both the French-German-Swiss scale and the S.D.C. Grey Scale No. 2 is, however, quite large, and represents a loss in depth of about 40–50% for patterns of normal depth. If, for example, a washing treatment removes nearly half the colour originally present, the pattern can scarcely be classified as having a good washing fastness, nor would the most tolerant assessor describe the pattern as only slightly altered.

The Committee, therefore, considers that Grade 3 should indicate a change much less than that illustrated in both scales, and that it should be approximately equal to the present Grade 4 of Grey Scale No. 2.

If this criterion is accepted, however, then it is clear that an equally spaced scale will not cover adequately the patterns of poor fastness. There are several methods of altering the spacing 3–2 and 2–1 to overcome this defect, but of these the most logical is to employ a geometric scale; that is to say, if the visual difference between original and treated patterns assessed as 4 is one unit, then Grade 3 represents a change of two units, Grade 2 four units, and Grade 1 eight units. On the geometric scale, the most important spacings 5–4 and 4–3 are, of course, visually equal.

In order to illustrate the principle, a geometric scale was prepared based on the application of the Adams "chromatic value" formula † for computing small colour differences. This formula was chosen because it is the easiest to apply and is quite accurate when, as here, no change in hue is involved. By using a factor $f = 40$ in this formula, the colour differences are expressed in "just perceptible steps" (j.p.s.). One j.p.s. is approximately equal to the Judd or N.B.S. unit, which has been set to indicate a colour difference of some commercial importance [1].

* Deutsche Echtheitskommission.

† Colour difference—
$\Delta E_A = f\{[0{\cdot}23\ \Delta V_y]^2 + [\Delta(V_x - V_y)]^2 + [0{\cdot}4\ \Delta(V_z - V_y)]^2\}^{\frac{1}{2}}$

Table I shows the spacing of this geometric grey scale and the spacing of the equally spaced scales.

TABLE I

Grade	Geometric Scale (j.p.s.)	Continental Scale (j.p.s.)	S.D.C. Grey Scale No. 2 (j.p.s.)
5	0	0	0
4	2·5	4·2	4·9
3	5	8·4	9·9
2	10	12·6	15·1
1	20	16·8	17·5*

* The bulk preparation of this scale gave too great a depth for Grade 1, but because of its relative unimportance it was not immediately corrected.

This geometric scale was considered by the International Standards Organisation at its meeting in Bournemouth in June 1951, and was accepted in principle. It was decided, however, to construct three additional geometric scales (Table II) to determine the most appropriate spacing. These scales have been sent to the Fastness Tests Committees of France, Germany, Switzerland, and the U.S.A., and it is hoped that international agreement with regard to the spacing will eventually be achieved.

TABLE II

Grade	Type A × 2 (j.p.s.)	Type B × 2 (j.p.s.)	Type C × 2·5 (j.p.s.)
5	0	0	0
4	2	1·5	1·5
3	4	3	3·75
2	8	6	9·5
1	16	12	24

The French-German-Swiss scale was designed to cover a range of depths of dyeing on the sliding-scale principle; however, as the original method of use has been abandoned in favour of a "contrast comparison" method, this complexity may be unnecessary, since it may be found that only two or even only one scale may be required for assessing loss in depth. This question can obviously be solved later, and the geometric scale can, if necessary, be extended to cover as many original depths of dyeing as the sliding equally spaced scale.

In America, the use of complex apparatus instead of the human eye for comparing two patterns is widespread, but it will probably be many years before such developments become general in Europe. The F.T.C.C. feels, however, that this need not cause differences in assessment, and if a visual aid in the form of a grey scale can be devised in co-operation with the A.A.T.C.C., world-wide agreement on this vitally important aspect of fastness testing may be reached.

* * *

The author wishes to acknowledge the assistance of Dr. I. M. S. Walls in the preparation and interpretation of the necessary spectrophotometric data.

DYEHOUSE LABORATORIES
IMPERIAL CHEMICAL INDUSTRIES LTD.
HEXAGON HOUSE
MANCHESTER 9

(*Received 29th March 1952*)

Reference

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PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE—VIII

The Use of the Grey Scale for Assessing the Effect on the Pattern in Fastness Testing

K. McLaren

It has been shown that a grey scale can be used for assessing not only loss in depth but also changes in hue and/or brightness and any combination of these three factors. A method is suggested for recording the effect on the pattern which requires the use of neither subjective terms nor fixed standards in different hues.

When a coloured pattern has been subjected to a fastness test, it is necessary to record how it has withstood the action of the destructive agency. To-day the usual method of doing this is to grade the pattern so that Grade 1 indicates a severe change in appearance and Grade 5 that no change has occurred; the degree of staining of adjacent undyed material is similarly graded, 1 indicating severe staining and 5 no staining. It is obvious, however, that the number must have a definite meaning which is understood by all concerned, and to-day the favoured method of eliminating all ambiguity is to employ fixed standards which illustrate the magnitude of each grade. The early systems employing fixed standards for assessing the effect on the pattern [1,2] were ambitious in that the standards were established in different hues, but the difficulties of accurate reproduction and of ensuring visual equivalence in each hue are very great; moreover, the possible permutations of simultaneous variation in the three attributes of colour—strength, hue, and brightness—and the fact that the original depth of dyeing in general fastness testing can vary over very wide limits would make adequate representation quite impossible. It was therefore a considerable advance when it was found [3] that, for assessing staining, the use of a single grey scale instead of scales in nine different hues did not result in any significant decrease in reliability.

To-day the concept of using a grey scale has extended to the assessment of the most common effect on the pattern in fastness testing, namely loss in depth [4]. This method was based on separately matching the original and treated patterns against a set of grey patterns ranging from black to white, the interval between the two being a measure of the degree of change. At the

Table I

Assessment of Patterns showing Mainly Changes in Hue by means of the Geometric Grey Scale

Assessor	Pattern A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T
1	4	3	4–5	3–4	4–5	4	3–4	4–5	1–2	3–4	3	3	4–5	1	4	3	1–2	4–5	4	4–5
2	4	3	4	4	5	4	3	4	2	3	3	3	4–5	1	4–5	2–3	1–2	4–5	3	4
3	4	3	4	3–4	5	4	3–4	4	2	3–4	3–4	4	4–5	1	4–5	3–4	2	4	3–4	5
4	4–5	2	4	3	5	3	3	3	1	3	3	3	3–4	1	4	2	1	4	3	4–5
5	3–4	3	4	3–4	5	4	3	4	2	3–4	3–4	3–4	4	1	4	3	2	3–4	3–4	4–5
6	3	3	4–5	3–4	5	4	3	4	2	4	3	3–4	3–4	1	4	3	2	3	3	4
7	4	2–3	4	4	4–5	4	2	3–4	1	3	3	3	3	1	4	2	1	2–3	3	4
8	4	2	4	3	4–5	4	2	3	1	3	2	2	3	1	4	2	1	3	3	4–5
9	4	3	4–5	4	4–5	4	3	4	1	3	3–4	3	4	1	4	2	2	3	4	4–5
10	4	3	3	3	5	3	3	3	2	3	4	4	4	1	3	2	2	4	4	4
11	4	3	4	4	5	3–4	3	4	2	3	4	4	4	1	4	2	2	3–4	4	4–5
12	4–5	3–4	4–5	3–4	5	3–4	3	4	3	4	4–5	4	4	1	4–5	3	2	4	4–5	5
13	4–5	2–3	4	2–3	4–5	3	2–3	3–4	1–2	3	3	3	3–4	1	4	2	1	3	3	4–5
14	4	2–3	4	3–4	5	4	3	3–4	2	3	4	4	4–5	1	4–5	3	2	4	4	5
15	3	2	4	3	5	4	2	4	1	3	4	4	4	1	4–5	2–3	1–2	4–5	5	5
16	4	2–3	4	3	4	3	2	2–3	1–2	3–4	4	3	3	1–2	4	2–3	2	3	3–4	5
17	4	3–4	4	4	5	3–4	3–4	4	3	4	4–5	4–5	4	2	4	4–5	3–4	4	4	5
18	4	3	4–5	2	5	4	3	4	1	2–3	5	3–4	4–5	1	2	2–3	1	3	4	5
19	4	3	4–5	4	5	4	3	4	2	3	3–4	4	4–5	1	4–5	3	2	4	4	5
20	4	3–4	5	4–5	5	4	4	4–5	3	3–4	3–4	4–5	5	1	5	3	1–2	4	4–5	5
21	4	2–3	4–5	2–3	4–5	3–4	3	3–4	1	2–3	3	4–5	4	1	4–5	2–3	1–2	4	4	5
22	4	3	4–5	4	5	4	2–3	4	2	3	3–4	4	4–5	1	4–5	2–3	1–2	3–4	3–4	5
23	4	4–5	4	3	4	4	2	4	1	2	3	4	3	1	4	4	1	3	2	5
Average assessment	4·0	2·9	4·2	3·4	4·8	3·7	2·8	3·8	1·7	3·2	3·5	3·6	4·0	1·1	4·1	2·7	1·7	3·6	3·7	4·7
Standard deviation	0·37	0·56	0·39	0·62	0·33	0·40	0·55	0·50	0·67	0·49	0·67	0·64	0·58	0·22	0·60	0·68	0·58	0·59	0·66	0·39

Average standard deviation (23 assessors, 20 patterns) = 0·52

international meeting in Basle in February 1951, however, it was agreed that a better method of assessing loss in depth was to equate the contrast between the original and treated patterns with the standard contrasts provided by a grey scale.

Subsequently it was considered that this new method of using a grey scale could also be used for assessing patterns which change in hue and/or brightness during testing. In order to verify this, twenty pairs of patterns showing changes in hue, strength, and brightness and combinations of two or all of these factors were assessed by twenty-three observers against a geometric grey scale. This scale consists of five pairs of grey patterns, the difference between the members of each pair increasing geometrically, viz. 0, 1·5, 3, 6, 12 just perceptible steps; the results are given in Table I. The average standard deviation for all assessments is 0·52, which compares favourably with 0·37 previously obtained in assessing stained patterns against a grey scale[3].

The Fastness Tests Co-ordinating Committee therefore suggests the following method for assessing every type of change which can occur in fastness testing (other than light)—

The original and treated patterns are laid side by side, and the difference between them is equated with the standard contrasts of the Geometric Grey Scale. If the difference lies between two contrasts of the Grey Scale, then half-grades are used, expressed in the form e.g. 3–4.

If the pattern has only lost depth, no further description is required. If, however, there is a significant change in hue and/or brightness, this is to be stated in qualitative terms, whilst an additional term is used if such a change is accompanied by a significant change in depth.

The following examples illustrate the application of this system—

Rating	Meaning	
3	Contrast equal to Grade 3 of the Grey Scale	Loss in depth is the only significant change
3 redder	Contrast equal to Grade 3 of the Grey Scale	There is no significant loss in depth but the pattern has become redder
3 weaker, yellower	Contrast equal to Grade 3 of the Grey Scale	Loss in depth and change in hue have occurred
3 weaker, bluer, duller	Contrast equal to Grade 3 of the Grey Scale	Loss in depth and change in both hue and brightness have occurred

Similarly a rating of "4–5 redder" would indicate that the pattern had become slightly redder, and a rating of "1 redder" that it had become very much redder, but that in neither case was there any significant change in brightness or depth.

The Committee thinks that in cases where two or three changes occur, it would be neither feasible nor necessary to indicate the relative magnitude of each change either in words (such as "much", "little", etc.) or by number (e.g. 1 = small, 3 = large), or even by putting the greater change first (i.e. "bluer, duller" being different from "duller, bluer").

It feels that the proposed method is preferable to any system which employs subjective terms, such as "small" and "large", for to provide illustrations of what constitute small, medium, and large changes of redness, blueness, yellowness, and dullness in a variety of original hues of different depth would be an impossible task; and without these indications, subjective terms are characterised by vagueness, especially when language differences exist.

It is realised that this proposal is subjective and therefore vague in one respect, viz. when does an observed change become significant? But this is not regarded as a serious defect.

The Committee would welcome comments on this proposal; it is hoped that the Geometric Grey Scale will shortly be available should anyone wish to examine the proposal in detail.

* * *

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Dyehouse Laboratories
Imperial Chemical Industries Ltd.
Hexagon House
Manchester 9

(*Received 29th March 1952*)

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COMMUNICATION

A Study of the Alkali Solubility Test for Determining the Degree of Fibre Damage in Wool

K. LEES and F. F. ELSWORTH

Existing methods for the determination of the alkali solubility of wool have been examined, especially in respect of their reproducibility. The physical state, e.g. yarn or fabric, in which the material is tested has been found to have considerable influence on the results. The effect of processes known to give rise to an increase in alkali solubility has been investigated with the object of giving significance to results on commercially processed materials. Examples are given of the use of the test in the examination of faults.

Introduction

It is known[1] that the solubility of wool in alkaline media is increased when it is treated with certain reagents which modify its chemical structure. Harris and Smith[2] showed that the degree of modification of wool arising from treatment with hydrogen peroxide or chlorine or exposure to light can be arbitrarily measured by determining the loss in weight when the wool is treated in an alkaline solution under standard conditions. Their method consisted in treating the wool in 0·1 N. sodium hydroxide at 65°C. for 1 hr., the liquor : wool ratio being approx. 100 : 1. They found that the solubility of wool which had not received any chemical treatment was approx. 10–13%. After treatment with hydrogen peroxide or chlorine or after exposure to a carbon arc its solubility had increased to an extent which depended on the severity of the treatment, e.g. strength of solutions and temperature or time of treatment. This change in alkali solubility was accompanied by a decrease in cystine content, the relationship between the two being shown to be linear for wool exposed to a carbon arc. If the treatment were sufficiently severe, the wool became completely soluble, this stage being reached with samples the cystine content of which was still at least 60% of that present in the untreated wool. Thus alkali solubility is a more sensitive measure of the modification of wool by oxidation than is the cystine content, and it was suggested that this is due to the fact that, although the main point of attack during oxidation is the disulphide group, other groups may also be attacked which affect the alkali solubility, without affecting the cystine content. Further, the resistance to wear of the samples treated with chlorine was found to decrease in a parallel manner to the increase in alkali solubility, but no similar data were given for wool treated with hydrogen peroxide or exposed to light. The authors claimed that the test provided a simple and convenient method of controlling processes which involve oxidation of the wool. It is now used as a test method in specifications controlling the production of shrink-resisting hosiery[3,4].

Ryberg[5] found that the solubility of wool when boiled in 0·05 N. sodium carbonate for 30 min. was of the same order as that obtained by Harris and Smith, and suggested that this method was more convenient for general laboratory use. In addition to obtaining similar increases in the solubility of treated wool as found by Harris and Smith, he found that the solubility of wool increased markedly as the result of damage by acid treatment. Since it can be inferred that the acid treatment would not have given rise to any change in the cystine content of the wool, Ryberg's results support the observation of the other authors that an increase in alkali solubility can also result from attack on groups other than the cystine linkage.

More recently, van Overbeke, Lefevre, and Raux[6] have studied systematically the effect of the various conditions, viz. concentration of reagent, time and temperature of treatment, and liquor ratio, on the solubility of untreated wool in sodium hydroxide solution. They found that the solubility increased in a roughly linear manner with increase in time and in sodium hydroxide concentration up to about 7 g./litre. Increase in temperature produced a slow increase in alkali solubility up to 60°C., but above that temperature the increase in alkali solubility was more rapid. As a result, these authors concluded that the optimum conditions for carrying out the test were treatment in 0·1 N. sodium hydroxide at 60°C. for 1 hr. with a liquor : wool ratio of 100 : 1. Apart from a small change in temperature, the method corresponds to that used by Harris and Smith.

The work now recorded represents a further examination of the alkali solubility test. The various methods previously described have been compared, especially in respect of the reproducibility of the results obtained, and the effect of the physical condition of the wool during the test has been studied in some detail. Finally the effect of processes known to give rise to an increase in alkali solubility has been investigated with a view to giving some significance to results obtained on materials which have been processed commercially.

Experimental

Unless otherwise stated, all tests have been carried out by the following methods—

The sample* was extracted with ethyl ether to remove fatty matter, and allowed to condition in the laboratory. Samples (1 g.) were then weighed

* The presence of acid in the samples under test will obviously reduce the effective strength of the alkaline solutions, and thus it is desirable that samples should be neutralised prior to test. An Australian specification[4] which uses the test prescribes that samples should first be steeped in sodium bicarbonate solution at 40°C. Nevertheless, the effect of amounts of acid normally present in wool (say up to 2%, calculated as sulphuric acid) will have but an insignificant effect at 100 : 1 liquor ratio on the strength of alkali. The Ryberg method, employing 0·05 N. reagent, will obviously be more sensitive to this factor than the other methods, which use 0·1 N. reagent.

out. One sample was dried to constant weight in an oven at 105° C., and its dry weight was used as the control weight of the remaining samples, which were subjected to the appropriate alkaline treatment as follows—

1. METHODS OF (A) HARRIS AND SMITH AND (B) VAN OVERBEKE, LEFEVRE, AND RAUX

A 250-ml. conical flask containing 100 ml. of 0·1 N. sodium hydroxide was placed in a water-bath maintained at 65 ± 1° C. (Harris and Smith method) or 60 ± 1° C. (van Overbeke, Lefevre, and Raux method). Rings of lead, placed around its neck, were used to sink the flask so that the level of the water was at least 1 in. higher than the level of the solution inside. The water in the bath was circulated vigorously by means of a centrifugal pump in order to ensure an even temperature distribution.

When the temperature of the sodium hydroxide solution had risen to the appropriate value, the weighed sample was introduced and the flask gently shaken to wet it out. The flask was then closed with a rubber stopper and allowed to remain in the water bath for 1 hr., after which the contents of the flask were poured quickly into a crucible with a sintered-glass filter, the alkaline liquor was removed by suction, and the sample was washed six times with distilled water, once with 1% acetic acid, in which it was allowed to remain for 2–3 min., and finally six times with distilled water. The crucible and contents were dried to constant weight, a small watch glass being placed over the crucible during the weighing. The alkali solubility is the loss in weight expressed as a percentage of the original dry weight of the sample.

2. METHOD OF RYBERG

The sample was placed in 100 ml. of 0·05 N. sodium carbonate solution which had previously been raised to the boil in a 250-ml. beaker, and treated at the boil for 30 min. The beaker and contents were heated on a controlled electric hot-plate, three pieces of broken pot being added, with the object of obtaining a regular rate of ebullition. The wool sample was recovered and weighed as in the other methods.

COMPARISON OF THE REPRODUCIBILITY OF THE METHODS

Preliminary experiments indicated that the rate of ebullition had a marked effect on the results obtained using Ryberg's method. Comparative tests were made on 2/32s worsted yarn (64s quality), cut into approx. ¼-in. lengths, using an old scratched beaker which boiled steadily and a new beaker which gave rise to erratic boiling with a tendency to "bumping" when heated on an electric hot-plate. The following mean results for the alkali solubility were obtained—

Using old beaker	...	15·1%
Using new beaker	...	17·9%

These results suggested that the Ryberg method was likely to be more difficult to control than the other methods using a lower temperature of treatment, and this was borne out by the results (Table I) obtained by the Ryberg, Harris and Smith, and van Overbeke methods on 2/32s worsted yarn cut into approx. ¼-in. lengths, which indicate that the reproducibility of tests carried out at the lower temperatures is of a higher order than those carried out at the boil. Difficulty in obtaining reproducible results when treating wool in boiling solutions has been experienced in these laboratories in connection with other work.

For these reasons the work described in the remainder of this paper has been carried out by the method of Harris and Smith except where otherwise stated. Much of the work had been completed prior to the publication of the method of Overbeke, Lefevre, and Raux, which gives results slightly lower than the Harris method, as is to be expected with the lower temperature used.

TABLE I

Method	Alkali Solubility (%) Results	Mean	Coefficient of Variation (%)
Ryberg	13·6 12·3 13·8 12·7 17·3 15·0 12·3 13·1 15·5 15·2 13·3 13·0	13·9 ± 0·44*	10·9
Harris and Smith	13·6 13·4 13·3 12·5 13·6 12·8 12·8 13·0 13·7 12·9 13·3 13·0	13·1 ± 0·1*	2·9
Van Overbeke *et al.*	10·6 10·6 11·6 10·9 10·7 10·5 11·2 10·7	10·8 ± 0·09*	2·4

* Standard error.

INFLUENCE OF PHYSICAL FACTORS ON ALKALI SOLUBILITY

Harris and Smith[2] drew attention to the influence on the alkali solubility of the physical state of the material; e.g. they found that a given sample of wool was more soluble as yarn than as fabric, but no results indicating the magnitude of the effect were quoted.

Valuable results using alkali solubility tests have been obtained from time to time in these laboratories in connection with the examination of faults and damages, and although tests of this kind must as far as possible be used on a comparative basis, it has been felt that the results obtained would in certain circumstances be of greater value if more information were available as to the effect of structural factors, thus facilitating their interpretation. This has now been the subject of a detailed investigation.

(*a*) *Wool Quality*

In the first instance a series of tests was made on four commercially processed wool tops of different quality. These results are given in Table II.

In considering these results, it must be borne in mind that the tops had not been processed under the same conditions. The results do not show any variation in alkali solubility which can be related to the differences in quality of the samples. They also suggest that the alkali solubility of typical grades of wool after commercial scouring is of the order of 12% when tested by the method of Harris and Smith and when the sample is in fibre form

TABLE II

Sample	Alkali Solubility (%) Results				Mean	Coefficient of Variation (%)
48s	12·5	12·6	12·3	12·3		
top	12·0	12·3	11·6	11·4	12·0 ± 0·14	4·0
	12·3	11·2	11·6	12·5		
58s	12·8	13·3	11·6			
top	13·9	10·0	13·5		12·5 ± 0·6	11·6
64s	11·7	11·5	10·9			
top	10·8	11·3	11·0		11·2 ± 0·15	3·2
70s	12·6	13·1	12·9			
top	13·3	13·0	12·1		12·8 ± 0·17	3·2

and thus free from the effects of spinning and conversion to fabric.

(b) *Yarn Twist*

Two lots of singles woollen yarn containing 22·2 and 7·5 t.p.in. were spun from the same wool blend. Samples of each yarn were treated together at the boil for 2 hr. in a solution maintained at pH 2 with sulphuric acid, rinsed, and dried. The alkali solubility of the yarns (½-in. lengths) before the acid treatment was determined by the Harris and Smith method and after the acid treatment by both the Harris and Smith and the Ryberg methods.

TABLE III

Method	Treatment	Twist	Alkali Solubility (%) Results				Mean	Coefficient of Variation (%)
Harris and Smith	Untreated	Hard	8·5	10·6	10·0	9·1	9·6 ± 0·5	9·1
		Soft	12·0	11·1	11·1	11·2	11·4 ± 0·22	3·8
Harris and Smith	Acid-treated	Hard	20·2	19·8	19·6	19·5	19·8 ± 0·16	1·6
		Soft	26·0	25·4	23·1	25·2	24·9 ± 0·6	5·1
Ryberg	Acid-treated	Hard	15·9	19·4	16·7	20·6	18·2 ± 1·11	12·2
		Soft	23·2	22·8	20·4	24·3	22·7 ± 0·82	7·3

The results (Table III) indicate that the harder the twist in the yarn the lower will be the alkali solubility, a difference which was not eliminated by the agitation inherent in the Ryberg method.

(c) *Weaving*

A sample of white worsted serge which had been commercially prepared for dyeing was treated at the boil for 3 hr. in a solution maintained at pH 2 with sulphuric acid, rinsed, and dried. The alkali solubility of the cloth both before and after acid treatment was determined in the form of approx. 1 in. or ½ in. squares and after dissection into ½ in. lengths of yarn.

The difference in the results (Table IV) is apparent on both the untreated and the acid-treated samples, but it is much more pronounced when the alkali solubility is high.

(d) *Knitting*

The 2/32s worsted yarn used for the comparison of methods was knitted into plain tubular fabric containing 20 stitches and 28 courses per inch. The fabric was cut into pieces approx. 1 in. square, and the alkali solubility determined.

The following results were obtained—

10·3, 8·8, 8·7, 10·3, 9·8, 8·8, 10·0, 9·8, 10·1, 10·0, 9·2

Mean 9·6 ± 0·19% Coefficient of variation 6·5%

The mean alkali solubility of the same wool in the form of ½ in. lengths of yarn was 13·1% (Table I), and thus there is a marked decrease in the alkali solubility as the result of knitting into fabric.

TABLE IV

Form of Sample	Alkali Solubility (%) Results				Mean	Coefficient of Variation (%)
	UNTREATED CLOTH					
1 in.	8·9	8·9	8·2	8·2		
squares	7·8	7·8	7·4	7·8	8·1 ± 0·14	6·1
	8·3	8·3	8·0	7·4		
½ in.	9·8	10·2	9·3	9·7		
squares	8·9	9·2	9·2	9·1	9·1 ± 0·23	8·4
	8·9	7·2	8·9			
½ in.	10·5	10·5	10·5	10·2		
lengths	11·4	11·7	11·5	11·7	11·2 ± 0·17	5·3
of yarn	11·4	12·0	11·3	11·2		
	ACID-TREATED CLOTH					
1 in.	18·0	17·7	17·9	18·7		
squares	20·2	18·6	18·3	17·5	18·4 ± 0·3	5·6
	18·4	20·7	18·1	16·4		
½ in.	26·0	25·2	24·7	26·2		
squares	24·5	27·4	26·2	26·2	25·6 ± 0·24	3·3
	25·4	24·9	25·6	24·9		
½ in.	41·1	40·7	40·6	41·7		
lengths	40·8	43·1	41·7	42·8	41·9 ± 0·34	2·8
of yarn	40·7	42·7	43·7	43·6		

General Observations

It is clear from the preceding experiments that the more compact the fibres as the result of spinning and weaving or knitting the lower is the alkali solubility obtained. The difference is most marked when the alkali solubility of the wool is high, and thus the effect is to decrease substantially the sensitivity of the test. For example, the tests carried out on the worsted serge (§ (c) above) show that the effect of the acid treatment is to increase the alkali solubility from 11·2% to 41·9% if the tests are made on the yarn, whereas tests made on 1 in. squares of cloth indicate only an increase from 8·1% to 18·4%.

Ideally it would appear that the most satisfactory method of carrying out the test would be to break the sample down to fibrous form and to maintain a continuous relative movement between the fibres and the liquor during the test, by either agitation or circulation. It is clearly impossible in a routine test to disintegrate each sample into fibre form, but as far as possible all samples tested in these laboratories are broken down into yarn fragments approx. ½ in. in length. The question of providing movement of the liquor during the test would probably repay further investigation.

EFFECT OF PROCESSING ON ALKALI SOLUBILITY OF WOOL

(1) *Peroxide Bleaching*

The effect of treating wool in hydrogen peroxide solutions is shown in Tables V and VI. These determinations were made on 2/32s worsted yarn (64s quality) which had been carefully scoured and treated with hydrogen peroxide in the laboratory in connection with other work.

TABLE V

Alkali Solubility (%) of Yarn after Treatment at 50°C. in Buffered Hydrogen Peroxide Solution

(Initial pH 9·6. Liquor : wool ratio 100 : 1)

Time of Treatment (hr.)	2-vol. H_2O_2 Results		Mean	5-vol. H_2O_2 Results		Mean
0	13·1 13·8	13·3 13·4	13·4	13·1 13·8	13·3 13·4	13·4
3	42·4 36·4	42·6 35·8	39·3	66·7	64·7	65·7
6	55·4 55·7	56·7 54·8	55·6	78·8 77·4	77·3 76·3	77·5
18	71·2 71·6	73·5 74·8	72·8	78·8 82·0	81·5 80·3	80·6

TABLE VI

Alkali Solubility (%) of Yarn after 3 hr. Treatment in Buffered Hydrogen Peroxide Solution

(Initial pH 7·6. Liquor : wool ratio 100 : 1)

Temperature of Treatment (°C.)	2-vol. H_2O_2 Results		Mean	5-vol. H_2O_2 Results		Mean
Untreated	13·1 13·8	13·3 13·4	13·4	13·1 13·8	13·3 13·4	13·4
30	16·1 15·7	15·6 16·3	15·8	16·8 16·3	16·7 16·5	16·6
50	25·8 27·5	24·8 26·6	26·2	45·0 46·1	42·8 45·1	44·8
70	61·0 60·6	61·8 56·5	60·0	83·7 —	81·3 —	82·5

These results indicate that the peroxide bleaching of wool at pH values above 7·6 causes an increase in its alkali solubility to an extent which is closely related to the severity of the treatment. The effect of the alkaline treatment itself on the alkali solubility can be neglected, since it is small and in the reverse direction. Thus yarn treated for 18 hr. at pH 9·6 in the absence of hydrogen peroxide, corresponding to the treatment given to the samples in Table V, had a mean alkali solubility of 8·5% after treatment. Hence, as indicated by Harris and Smith, the test provides a reasonably sensitive measure of the effect of hydrogen peroxide treatment, and can be used to assess the severity of processing. This is well illustrated by the results obtained on the same yarn which had been bleached commercially in bulk by six different bleachers (Table VII).

TABLE VII

Alkali Solubility (%) of Yarn treated in Commercial Peroxide Bleaching Processes

Sample	Results				Mean
Untreated	13·1	13·3	13·8	13·4	13·4
1	16·8	16·8	16·4	16·6	16·6
2	23·5	23·5	23·7	22·7	23·4
3	33·7	36·7	36·2	36·2	35·7
4	51·7	53·6	54·4	48·5	52·1
5	35·6	34·7	34·9	36·4	35·4
6	61·4	62·4	57·9	60·7	60·6

(2) *Carbonising*

The effect of carbonising on alkali solubility was first studied on a series of samples prepared in connection with other work. Samples of knitted fabric made from 2/14s worsted yarn spun from the same tops as the yarn used for the peroxide bleaching tests were steeped in sulphuric acid solutions of different strength, hydroextracted, dried, and baked. Apart from the variation in the strength of the acid solutions, all other conditions were kept constant throughout. The samples were well rinsed, carefully neutralised to pH 6 using dilute sodium carbonate solution at room temperature, and their alkali solubilities determined. The acid content of the samples before neutralising was determined by the pyridine method [7].

TABLE VIII

Alkali Solubility (%) of Wool after Carbonising

Acid Content after Carbonising (% as H_2SO_4)	Results		Mean
Untreated	12·4 12·9	12·3 12·8	12·6
3·7	14·5	14·6	14·6
5·8	15·9	15·6	15·8
7·4	17·4	17·2	17·3
10·6	24·5	24·4	24·5

The results (Table VIII) indicate that carbonising results in an increase in alkali solubility. In relation to commercial practice, however, the results are more difficult to interpret than with peroxide bleaching, since it is difficult to reproduce on the laboratory scale the drying and baking conditions employed in full-scale carbonising, and the effects of laboratory variations of these factors have not yet been studied.

Some determinations were made on woollen pieces (blankets) before and after they had been carbonised and neutralised commercially. The following results were obtained—

Scoured only	11·4	12·0	10·5	10·3	11·5	10·3
After carbonising and neutralising	14·0	13·6	13·8	15·1	14·2	13·3

Only a small increase in alkali solubility has taken place, but it must be borne in mind that the pieces had been fully neutralised to a pH of the order of 9. Some alkali-soluble material may have been removed during this treatment, thus giving rise to low results.

The results obtained on both the laboratory and commercially carbonised samples suggest that the test is less sensitive as a measure of the effect of the carbonising process than of peroxide bleaching, since it would appear that the upper limit for the alkali solubility of wool, initially in sound condition, after normal carbonising processing would be of the order of 20%, whereas values up to 60% have been obtained on commercially peroxide-bleached wool (Table VII). Some evidence has been obtained that the alkali solubility of carbonised wool increases rapidly if it is allowed to remain in an acid condition. Thus the alkali solubility of a carbonised woollen flannel was found to increase from 34% to over 80% on standing for four months.

The test has given results of value in the examination of dyeing faults believed to arise as the result of uneven treatment in carbonising.

Some examples of the use of the test in this way are given below.

(*a*) A woollen piece which had been scoured, milled, carbonised, and dyed, after which it contained a stripe running roughly in the direction of the warp, which was lighter in shade than the bulk of the cloth. Stripping with ammonia and redyeing with Methylene Blue 2BS gave the dyeing reversal which is typical of this type of fault [8]. Alkali solubility tests made on samples taken from the light and dark areas gave the following results—

Light	...	18·4 19·3	(mean 18·8%)
Dark	...	14·7 14·7	(mean 14·7%)

thus suggesting that the light area had received a more severe acid treatment.

(*b*) A blue carbonised and piece-dyed blanket cloth which contained a broad stripe in which the material was both lighter in shade and very tender. The cloth within the stripe was very lean, which suggested that the damage was so severe that considerable wastage of fibre had already occurred. On stripping with ammonia and redyeing with Methylene Blue 2BS, the light stripe dyed much darker than the remainder of the piece. Alkali solubility determinations made on the two portions of the cloth gave the following results—

Light	...	22·6 22·3	(mean 22·5%)
Dark	...	12·3 13·1	(mean 12·7%)

Again uneven acid treatment is indicated, but the result is obviously low, taking the condition of the material into account. It would appear that much of the wool substance rendered soluble as a result of carbonising had already been removed in subsequent processing, thereby giving rise to low results on both parts of the piece.

(*c*) Shade differences were found between individual hanks of woollen yarn which had been spun from carbonised wool, scoured, and dyed in hank form. After stripping in ammonia and redyeing with Methylene Blue 2BS the light hank dyed more darkly. Alkali solubility tests on the two samples gave the following results—

Light hank	...	22·2 23·2	(mean 22·7%)
Dark hank	...	18·1 17·2	(mean 17·7%)

(3) *Chlorination*

Worsted yarn (2/32s, 64s quality) in hank form was chlorinated in the laboratory by both wet and dry processes. For dry chlorination, four hanks were given 0·5, 0·9, 1·1, and 1·4% chlorine respectively on the weight of wool, after which they were neutralised by treatment at room temperature in a solution containing 5% sodium sulphite on the weight of the wool, and then rinsed and dried.

For the wet chlorination the hanks were treated in hypochlorite solutions acidified to pH 2 with hydrochloric acid and containing 1, 2, 3, and 4% chlorine respectively on the weight of the wool. The necessary quantities of sodium hypochlorite and hydrochloric acid were added dropwise from a burette over a period of 30 min. with constant stirring. The samples were then neutralised with sodium sulphite as described above.

Samples of the knitted fabric made from the same yarn have also been treated commercially by four different shrink-resistant finishes all involving the use of chlorine.

The alkali solubility of the various samples in yarn form has been determined, and the results are given in Table IX together with the area shrinkage (felting) when the samples were tested according to the method used on utility hosiery fabric [9].

Table IX

Process		Chlorine (%)	Alkali Solubility (%) Results				Mean	Area Shrinkage (%)
Untreated		—	13·1	13·3	13·8	13·4	13·4	14·1
Wet chlorination		1·0	18·8	19·1	18·3	—	18·7	7·1
		2·0	23·5	21·8	21·4	20·7	21·9	7·9
		3·0	22·9	22·9	22·2	22·4	22·6	4·3
		4·0	31·4	32·3	32·0	31·7	31·9	−2·6
Dry chlorination		0·5	16·6	16·3	16·5	17·0	16·6	12·5
		0·9	16·9	17·2	17·4	16·6	17·0	7·4
		1·1	17·5	17·5	16·5	17·3	17·2	6·2
		1·4	18·1	18·1	18·3	18·2	18·2	5·8
Commercial shrink-resistant processes	A	—	20·8	20·8	21·9	21·2	21·2	5·5
	B	—	17·5	16·8	18·9	19·3	18·1	1·9
	C	—	22·7	23·2	24·8	25·4	24·0	1·8
	D	—	18·7	18·4	19·1	18·3	18·6	4·1

The results show that chlorination causes an increase in the alkali solubility of wool. At chlorine concentrations normally employed in commercial processing the results on both the laboratory and commercially treated samples suggest that this increase is small. There is, however, an indication that overtreatment in wet chlorination will result in a substantial increase in solubility. It is of interest to note that in certain specifications [10] controlling the production of shrink-resisting wool materials the difference between the alkali solubilities of the treated and untreated material must not exceed 6%.

(4) *Boiling in Acidic Solutions*

Worsted yarn (2/14s) in hank form was treated in solutions of different acidity, the treatment being carried out in a small pressure dyeing machine. The treatment was begun with the solution at 40°C., and after bringing to the boil in 50 min. was continued at the boil for a further 3 hr. An appropriate amount of sulphuric acid was added to each solution in order to bring it to the pH chosen for the boiling treatment. Small additions of acid were made from time to time, if necessary to maintain these conditions. After thorough rinsing in warm water the yarn was neutralised by overnight steeping in dilute pyridine solution followed by further washing and drying, and the alkali solubility of the treated yarn was then determined.

The results (Table X) indicate that the treatment of wool in acid solution gives rise to an increase in alkali solubility. This increase is small under conditions which would normally be met with in acid dyeing processes. Thus the treatments at pH 2·8 and 3·4 represent conditions of acidity which correspond to those employed in dyeing with level dyeing acid dyes, but are probably more severe in

TABLE X

pH of Treatment	Alkali Solubility (%) Results		Mean
Untreated	12·4	12·3	12·6
	12·9	12·8	
1·5	66·8	66·1	66·5
2·2	34·8	35·2	35·0
2·8	19·4	20·1	19·8
3·4	16·6	16·7	16·7
6·0	12·8	13·0	12·9

respect of time and temperature. They thus indicate an upper limit for the alkali solubility of wool, initially in a sound condition, after dyeing. A substantial increase in alkali solubility results from treatment at pH 2·2 and 1·5. These conditions are more severe than those normally met with, although dyeing wool with an addition of 6–8% sulphuric acid on its weight would give a dyebath pH at least as low as 2·2.

Two further examples of the use of the test in connection with the examination of faults may be given—

(*a*) A woollen piece, which had been scoured, milled, carbonised, and dyed, was found to be very tender. Other pieces of the same quality were satisfactory after the same processing. Alkali solubility tests were carried out on the cloth in the loom state and after dyeing.

TABLE XI

Sample	Alkali Solubility (%) Results		Mean
Cloth in loom state ...	16·4	16·3	16·4
	16·3	16·7	
Tender cloth	40·1	37·5	38·8
Satisfactory cloth ...	24·0	24·7	24·4

The results (Table XI) clearly indicate a difference in the severity of the processing which the two pieces had received. Both dyed pieces were in an acid condition, the pH of a water extract (1 g. in 50 ml.) being 2·8, and therefore must have been dyed in a dye liquor having a pH at least as low as this. The difference in alkali solubility therefore suggests that the tender piece had received a prolonged dyeing treatment.

(*b*) Complaints were received with respect to the wearing properties of girls' gymnasium slips made from a worsted serge and piece-dyed green with acid dyes. Garments made from the same cloth dyed navy blue by the afterchrome method gave no cause for complaint. The results in Table XII were obtained by determinations made on the two cloths after dyeing.

TABLE XII

	Green	Blue
Alkali solubility, %	28	16
Abrasion resistance (No. of rubs necessary to wear holes using Martindale abrasion tester [11])	2440	8705
pH of water extract (1 g. in 50 ml.) ...	3·2	6·7

The results of the abrasion tests show a pronounced difference in the performance of the two samples, and this and other work concerned with the effect of abrasion and chroming of wool indicates that the effect due to chrome would be much less than the difference now found. The alkali solubility results reflect this difference, and suggest that the green cloth has received an excessive treatment in an acidic dye liquor.

Discussion

The determination of damage in wool is a complex problem, especially when it is desired to translate results into an assessment of the performance of the material in consumer use, e.g. in wear or in washing. Because of its chemical composition and especially because of the rôle played by the covalent disulphide cross-linkages, the degradation of wool by chemical and other agencies such as heat and light occurs as the result of at least two reactions, acting independently or together, viz. breakdown of the disulphide linkages themselves and hydrolysis of the main peptide chains. The behaviour of peroxide-treated wool on the one hand, and of wool boiled in acid solutions on the other, shows that both reactions result in an increase in the alkali solubility.

The results presented in the preceding sections indicate the magnitude of the increase in the alkali solubility when wool is subjected to chemical treatment similar to that involved in four different finishing processes. They show that the results of alkali solubility tests can be adequately interpreted only if the treatment to which the sample has been subjected is known or can be safely presumed. Thus it has been shown that a dyed worsted cloth with an alkali solubility of 28% gave a most unsatisfactory performance in actual wear. In contrast, four out of the six commercially peroxide-bleached samples examined (Table VII) had an alkali solubility in excess of this value, and it would seem, therefore, that an alkali solubility greater than 30% is not unusual in commercial peroxide bleaching. These and other results suggest that attack on the disulphide linkage results in a more pronounced change in alkali solubility than peptide chain hydrolysis.

Certain treatments which result in cross-linkage rebuilding, e.g. formaldehyde, chrome dyeing, are known to decrease the alkali solubility of wool. Hence aftertreatments by these reagents may result in an alkali solubility which would not then be indicative of the amount of damage caused in an earlier process.

When an increase in alkali solubility is not accompanied by a decrease in cystine content, it can be safely presumed that the damage results from peptide chain hydrolysis during processing under acid conditions. On the other hand, a simultaneous decrease in cystine content indicates that the wool has received a treatment involving oxidation.

No attempt has been made in the present work to correlate alkali solubility with the behaviour of chemically processed materials in consumer use, but it is hoped to investigate this more fully in the future.

* * *

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WOOL INDUSTRIES RESEARCH ASSOCIATION
TORRIDON
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Notes

Proceedings of the Council

At a meeting of the Council held at the Victoria Hotel, Bradford, on 9th April 1952, the proceedings included the following items of interest—

ELECTION OF CHAIRMAN— Mr. H. H. Bowen (*President of the Society*) was elected Chairman of the Council for 1952–53.

ELECTION OF VICE-CHAIRMAN — Mr. F. L. Goodall was re-elected Vice-chairman of the Council for 1952–53.

NEW MEMBERS OF COUNCIL— The President extended a cordial welcome to new members of the Council (cf. J.S.D.C., **68**, 149 (May 1952)). It was reported that the following had recently become *ex officio* members of the Council—

Mr. R. D. Alexander (*Chairman, Scottish Section*)
Mr. S. L. Peel (*Chairman, West Riding Section*)
Mr. E. Rolfe (*Chairman, Huddersfield Section*)
Mr. G. F. Wood (*Chairman, London Section*)

MEMBERSHIP COMMITTEE— It was resolved to set up a Membership Committee, to consist in the first instance of the Honorary Secretaries of Local Sections and Junior Branches, to consider methods of increasing the membership of the Society.

PUBLICATION OF RESULTS OF POSTAL BALLOTS— It was resolved that in future the numbers of votes cast for both successful and unsuccessful candidates would be published.

MEMBERSHIP OF COMMITTEES— The membership of non-technical committees was reviewed for the ensuing year. The resignation of Mr. A. Breare from the Publications Committee was accepted with regret, and the retirement of Mr. R. J. Hannay in order of seniority from the Dyers' Company's Medal Committee was noted.

REPRESENTATION ON EXTERNAL BODIES— The representation of the Society on external bodies was reviewed for the ensuing year. The resignation of Dr. R. L. Elliott from the Textile Institute Unification of Testing Methods Committee was accepted with regret.

HONORARY SOLICITORS— It was resolved to invite Messrs. John Conchar & Co. to serve as Honorary Solicitors for the ensuing year.

HONORARY PATENT AGENT— It was resolved to invite Mr. L. E. Jones to serve as Honorary Patent Agent for the ensuing year.

MEMBERSHIP— Twelve applications for ordinary membership and two for junior membership were approved. Mr. J. N. Dutfield was readmitted to membership of the Society.

Meetings of Council and Committees
May

Council— 14th
Finance— 14th
Publications— 20th
Colour Index Editorial Panel— 28th
Fastness Tests Co-ordinating— 12th
Terms and Definitions— 9th
Printing Symposium— 8th
New Diploma Committee—27th

A.G.M. and Dinner 1953

Arrangements have been made to hold the Sixty-ninth Annual General Meeting and Dinner on Friday, 10th April 1953, at the May Fair Hotel, Berkeley Street, London W.1.

Annual Meeting of Chairmen and Honorary Secretaries of Sections 1952

This meeting was held at the Queens Hotel, Leeds, on 29th March 1952, under the chairmanship of Mr. H. Jennison (Honorary Treasurer of the Society). Topics which were discussed included suggested subjects for lectures in the 1952–53 session, publication of Junior Branch and Summer School lectures, membership, and the new Byelaws relating to Vice-presidents.

Fastness to Cellulose Ester Bonding

In the Second Edition of the *Second Report of the Fastness Tests Committee* (May 1950), it was stated that the Cellulose Ester Bonding Test was under revision in view of recent changes in bonding practice. The Fastness Tests Co-ordinating Committee has now considered this test in all its aspects, and has decided that the existing test should be abandoned and not replaced. The main reasons which influenced the Committee in its decision not to recommend an alternative test were, firstly, that reliable results can be obtained only if patterns are tested in a press, the domestic iron being unsatisfactory, and secondly that there is always a possibility of further changes in bonding practice being made.

Production of Synthetic Dyes begun in India

Production of synthetic dyes at the Bulsar (near Bombay) plant of Atul Ltd. started in April 1952. This company, 10% of whose capital is owned by the American Cyanamid Co., the rest being Indian, intends to produce a range of twenty-five of the most commonly used dyes, the first to be made being sulphur dyes. C O C

Vitron—Continuous-filament Glass Fibre

Vitron is a continuous-filament glass fibre now being produced in the U.S.A. by Glass Fibers Inc. C O C

Technical Information and Documents Unit

The Headquarters Technical Information Service of the Department of Scientific and Industrial Research has been merged with the Technical Information and Documents Unit of the Department, and will in future be known by the latter name. Its address is now Cunard Building, 15 Regent Street, London S.W.1 (telephone WHItehall 9788). The Unit maintains a technical enquiry service, provides information about industrial techniques from America and certain European countries, and receives unpublished reports from British and American sources. A reading room is open on weekdays and Saturday mornings.

American Library—Industrial Section

The American Library, at 41 Grosvenor Square, London W.1, has opened an industrial section, which includes American books, periodicals, and Government documents on technology and management. Books, etc. may be borrowed by anyone in Great Britain, and will be sent by post. Photocopies of special articles will be provided, and the library also answers requests for information by letter or telephone (GROsvenor 9000, Extension 447 or 748). The library acts as a liaison with appropriate agencies in the U.S.A. where information in its own collection is deficient. The library is open to the public Mondays–Fridays, 9 a.m.–6 p.m., and all services are free.

Centre d'Information de la Couleur

A Colour Information Centre has been formed in Paris by AFNOR (Association Française de Normalisation). The Centre has been set up to give information to all persons interested in the problems of colour in its widest aspects. The Centre will play a co-ordinating rôle by the establishment of a central index kept up to date as it receives advice of new work announced, new research undertaken, and above all new results obtained, and progress achieved in the increasing knowledge of the world of colour.

New Books and Publications

Die neuzeitlichen Textilveredlungs-Verfahren der Kunstfasern
5. Lieferung

By F. Weber and A. Martina. Pp. 647–786 + i–xx. Vienna: Springer-Verlag. 1951. Price, 41s. 0d.

This completes a most useful index to the patents taken out during 1939–1950 on the finishing of man-made fibres. This instalment contains the sections on—Detergents and Washing Processes (pp. 647–689), Milling (690–693), Carrotting (693–696), Chlorination of Wool (696–700), Lubrication and Conditioning (700–713), Carbonising (713–715), Kier-boiling (715–716), Crabbing and Setting (716–718), Crêping (718–722), Yarn Sizing (723–733), and Mercerising (733–742), together with a Subject Index to the completed work and a Patent Index. The latter is arranged numerically under the different countries, which however are not given alphabetically but haphazardly. There is no author index, and the lack of it will be felt by those using this publication. For each section a selected list of literature references is given, but no selection has been shown in the listing of the patents, many being included which are most unlikely to find application on fabrics containing man-made fibres or which deal almost entirely with the manufacture of a particular agent; e.g. most of the patents under the detergent section deal with the manufacture of detergents and not with novel methods of using them. Sections such as those on milling and chlorination are included because fabrics containing artificial fibres may be subjected to those processes, but why the sections on carrotting and carbonising were included passes understanding; they are lost in such a work, for no one desiring such information would ever think of consulting a work dealing with the finishing of artificial fibres. However, if the authors have included too much, it cannot be said that they have made any serious omissions. The book will long remain a standard work of reference. C. O. CLARK

Six-membered Heterocyclic Nitrogen Compounds with Four Condensed Rings

By C. F. H. Allen and Collaborators. Pp. xiii + 345. New York and London: Interscience Publishers. 1951. Price, $10.00 or 80s. 0d.

This is the second volume to appear in the series of monographs devoted to the chemistry of heterocyclic compounds, and fully maintains the very high standard set in the volume written by Dr. F. G. Mann. It is a co-operative work, from the Eastman Kodak laboratories, to which six individual authors have contributed the various chapters and subsections; yet the whole book reads smoothly and the style is consistent throughout. From its title the book may be considered as dealing with a relatively narrow field of chemistry, but it is one of great complexity and of considerable technical as well as academic interest. Moreover, as workers in this field know full well, information on the compounds discussed is scattered widely over the academic and technical literature, a large proportion of it being available only in the form of patent specifications, and a literature search is made even more difficult than usual by the great

variety of systems of nomenclature in use by different investigators.

The "aza" nomenclature is quite rightly preferred in this book, and names of particular compounds are usually followed by the *Chemical Abstracts* systematic name, the *Ring Index* number, and where necessary other names which have become current in the literature. This is a great help to the reader, and this clarification alone would make this literature survey worth while for the student and the research worker with little or no experience of this type of compound. Quite apart from the casual student, however, it should be of real value also to the expert as a very complete survey up to the end of 1950, and is a valuable addition to chemical literature. The book will interest especially two main groups of chemists interested respectively in dyes and natural products. It has seven main chapters on aza analogues of naphthacene, 1 : 2 - benzanthracene, benzophenanthrene, chrysene, triphenylene, benzanthrene, and pyrene respectively. Chapter I on azanaphthacenes is of general interest and deals mainly with academic investigations. The chapter on azabenz[*a*]anthracenes is of particular interest to dye chemists and contains amongst other things accounts of dyes of the Alizarin Blue S type, the Skraup and Schmidt reactions in their special significance to the class of compounds under discussion, the vat yellows from benzoylated indigo, and compounds related to riboflavin. This chapter is not quite complete, however, because the 7- and 12-azabenz[*a*]anthracenes will be discussed in a subsequent monograph devoted to derivatives of acridines. Chapters III (azabenzo[*c*]phenanthrenes), IV (azachrysenes), and V (azatriphenylenes) are not concerned with dye chemistry but with natural products, including some of the *iso*quinoline alkaloids. The most interesting section, comprising one-third of the book (Chapter VI and addendum (pp. 311-314)), deals exhaustively with the azabenzanthrenes and so includes the anthrapyridine, anthrapyridone, and anthrapyrimidine dyes as well as the aporphine alkaloids. The final chapter deals with the azapyrenes and includes the diazapyrene dyes (Indanthren Scarlet 2G and Brilliant Orange GR).

The book is excellently presented and singularly free from errors, although perhaps the penultimate paragraph of p. 280 may be a little misleading, since the effect of an amino group on colour in the anthrapyrimidines is much more pronounced in the 8- and 11-positions than in the 6-position. It is well printed and has a very comprehensive index. The only objection which may be raised is the relatively high price to British chemists.

S. Coffey

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University.

Imperial Chemical Industries Ltd.

Dyestuffs for Full Chrome Suède Calf Leather—This pattern card contains forty-four dyeings of homogeneous dyes, including normal acid dyes, dyes from the Naphthalene Leather range, and a direct black diazotised and developed with *m*-toluylenediamine. Details of the dyeing method employed, solubility, penetration, and light fastness are appended.

Waxoline Dyestuffs—This card illustrates a range of twenty-four Waxoline dyes. They are divided into two groups—Type A, directly soluble in oils, fats, waxes, fatty acids, and common solvents; and Type B, free bases which, owing to their poor solubility in all but fatty acids, are dissolved in the latter, the resulting fatty acid compounds being used for colouring oils, etc. Details of the solubilities of each dye in particular solvents are given, and notes on special properties and uses are appended.

Acid Milling Dyestuffs on Wool Yarn—This card contains eighty-three dyeings on wool yarn, forty-four of which are mixtures. The text includes details of the dyeing methods employed, with special reference to the use of Carbolan Salt A as a levelling agent. Fastness properties quoted include light, perspiration, and repeated washing, and notes are also included on the general properties and uses of each dye.

Fixanol P in the Paper Industry—This pattern card includes samples of sized paper coloured with fourteen acid dyes. Each dye is illustrated by a pair of samples, one of which was produced using Fixanol P. The greatly increased depth obtained with this product is immediately obvious. The remaining thirteen colourings are on absorbent paper and are produced with acid dyes, which can be used if Fixanol P is added to the stock. Fastness figures appended include light, alkali, water, and bleaching.

Abstracts from British and Foreign Journals and Patents

(*The titles of abstracts may be modified. Abbreviations of names of firms are listed in* J.S.D.C., **68**, *23 (Jan. 1952); while other abbreviations and symbols, together with a list of the periodicals abstracted, will be found at the end of the annual index.*)

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Fibre-scouring and -washing Bowl. E. H. Thompson and E. Brookes. *BP* 668,579

A machine for scouring loose fibres comprises a tank or series of tanks each having three hollow perforated rollers, through which suction can be applied and which dip just below the surface of the scouring liquor. Alternating with these are three smaller breaker rollers. All the rollers are covered with curved plates which allow a small clearance and which are fitted with pressure plates for controlling and relieving pressure from excessive water or material on the suction roll. Transverse baffle plates under the suction rollers serve to provide, in effect, a number of separate washing zones as the material passes from one suction roll to the next. Fibre matting is avoided, excellent penetration is provided, and all dirt falls through spaces in the baffles to the base of the main tank.

J. W. B.

Applying Liquids to Airborne Fibres. Monsanto. *USP* 2,568,499

Continuous Dry Cleaning and Degreasing of Fibrous Material. M. T. Hoffman. *USP* 2,567,938

Apparatus for dry cleaning and depitching raw wool.

C. O. C.

Thread-advancing Reel having Spray and Drain Devices. H. A. Kuljian. *USP* 2,573,578

Hank-dyeing Machine. H. Nielsen. *BP* 668,018

The hanks are suspended on sticks, which are fixed in a cage so that they surround a reversible propeller placed in the centre of the dye tank. This arrangement gives very even dyeing. C. O. C.

Yarn-dyeing Machine. F. C. Neu. *USP* 2,573,047

A machine which secures even treatment of the material. C. O. C.

Treating Spools of Yarn by Liquid under Pressure. J. Schlumpf. *BP* 668,506

The spools are placed each on a perforated spindle, rigid plates being placed between the spools. Complementary conical seatings into which the spindles fit are placed between the ends of the spindles and the plates. The apex angle of these seatings is not > 45°, and the taper of the spindles differs slightly from the taper of the seatings associated with the plates. On the assembly being pressed together, an effective seal is formed between the spools and plates, there being no tendency for the liquor to leak between the spindles and the plates. C. O. C.

Automatic Control of the Consistency of Size. Dominion Textile Co. *USP* 2,572,351

Sizing and Drying Warps. McConnell Sales & Engineering Corpn. *USP* 2,572,288

Uncurling the Curled Edges of Fabrics. BrC. *BP* 669,421

The fabric is folded down its length with the face on the inside, and one run of a driven, endless flexible belt passed between and in frictional contact with the fabric faces at the curled edges, with a motion having a component transversely outwards in relation to the curled edges. The fabric is moved continuously lengthways and past the run of the belt, the edges of the fabric after being uncurled by the belt being retained uncurled by keeping the fabric faces pressed together. C. O. C.

Heating Continuously Moving Webs. Selas Corpn. of America. *USP* 2,571,425

The web may be moved to and from the heating chamber, e.g. of a drying or singeing machine, without altering the tension in the web or the length of the web extending between the operating parts of any machine with which the heating chamber co-operates. C. O. C.

Wet Processing Machines. Freeman, Taylor Machines. *BP* 668,894

A tank for holding the liquor and the goods has one or more ducts or passages, connected with its top and bottom or opposite sides or ends, and a rotary device within or adjoining the ducts or passages to circulate the liquor through the goods. The following advantages are claimed— high volumes with relatively low velocities through the pump, minimum change in the sectional area of the moving body of liquor, ability to obtain strong liquor movement against heads greater than could be dealt with by screw propellers, absence of agitation, low impeller speeds, reversibility of flow by reversing rotation of impellers, and minimum power losses on account of low operating speed and the positive movement of the liquor. C. O. C.

Piece-scouring Machine. T. Bailey. *BP* 669,109

A machine which can be used for both open-width and rope scouring. C. O. C.

Electrically Heated Drying Cylinder. M. Doniak. *USP* 2,571,426

Drying Cylinders. Pusey & Jones Corpn. *BP* 668,025

An improved means of removing condensate from steam-heated cylinders. C. O. C.

Cylinder Drying Machine. J. W. W. Evans. *BP* 668,738

The cylinders are mounted so that the geared drive has unvarying mutual engagement despite variations in the lengths of the cylinders due to changes in temperature and so that there is adequate sealing at the joints of the doll-head at all temperatures and working pressures. There is no tendency for the gear wheels to splash oil on to the cylinders. C. O. C.

Tenter. J. Dungler. *BP* 668,294

The gripping members are fixed on the outer side of a flexible inextensible endless band placed on edge. The weight of this band and the grips is supported by a set of spaced horizontal rollers, the transverse pull of the cloth under treatment being taken up by a vertical race on a fixed guide rail, a set of rollers carried by the endless band rolling on the vertical race. This gives smooth high-speed running with little wear; in addition, risk of oil stain on the cloth is obviated, while high temperatures can be used without troubles caused by loss of lubricant by evaporation. C. O. C.

Drying Web-like Material. A.-B. Svenska Fläktfabriken. *BP* 668,741

A machine in which the material moves in a horizontal plane is described. It has high efficiency, and in some cases the web can be supported and conveyed by the air used to dry it. C. O. C.

Drum Drying Machine. H. J. Preller. *BP* 668,157

Automatic clearance of fluff from the screens or filters in the air conduits is described. C. O. C.

Drying Flat or Tubular Knit Fabric. Samcoe Holding Corpn. *USP* 2,570,318

A tensionless drier which keeps the fabric unwrinkled. C. O. C.

Warp-printing Machine. H. Epstein. *USP* 2,573,097

A machine for the multicolour printing of warps with great accuracy of registration at high speeds. C. O. C.

Cloth-laying Machine. L. Spinelli. *USP* 2,570,877

A machine for quickly and easily applying cloth to a printing table so as to preclude wrinkling and creasing. C. O. C.

Mounting for Stencil Screens. C. F. Schaefer. *USP* 2,571,064

The stencil is mounted on a hollow drum, the printing paste being forced through the stencil from the interior of the drum, which rotates in contact with the material to be printed. C. O. C.

Reproduction of Coloured Pictures, etc. C. W. Jacob. *USP* 2,573,143

Apparatus for the inexpensive, simple, direct, and rapid reproduction of coloured pictures, etc. C. O. C.

Automatically Controlled Dry Cleaning Machine. Detrex Corpn. *USP* 2,574,251

A machine which has great flexibility of operation combined with automatic controls for carrying out a predetermined cycle of operations. C. O. C.

Laundry Ironing Machine. Machinefabriek Reineveld N.V. *BP* 668,255

Power-operated Flat Iron Press. Prosperity Co. *BP* 669,005

Extracting Moisture from Leather and Skins. Turner Tanning Machinery Co. *BP* 668,566

A rotary sammying machine is described in which the skin is squeezed from end to end in one continuous operation. C. O. C.

Fur Carrotting. J. F. Ellis. *USP* 2,572,125

A machine in which the carrotting liquor is brushed on to the skins. C. O. C.

Electrostatic Coating. Harper J. Ransburg Co. *BP* 669,978

Coating Paper (XI p. 230).

Sizing and Drying Paper as it passes to Roller Printing Machines (XI p. 231).

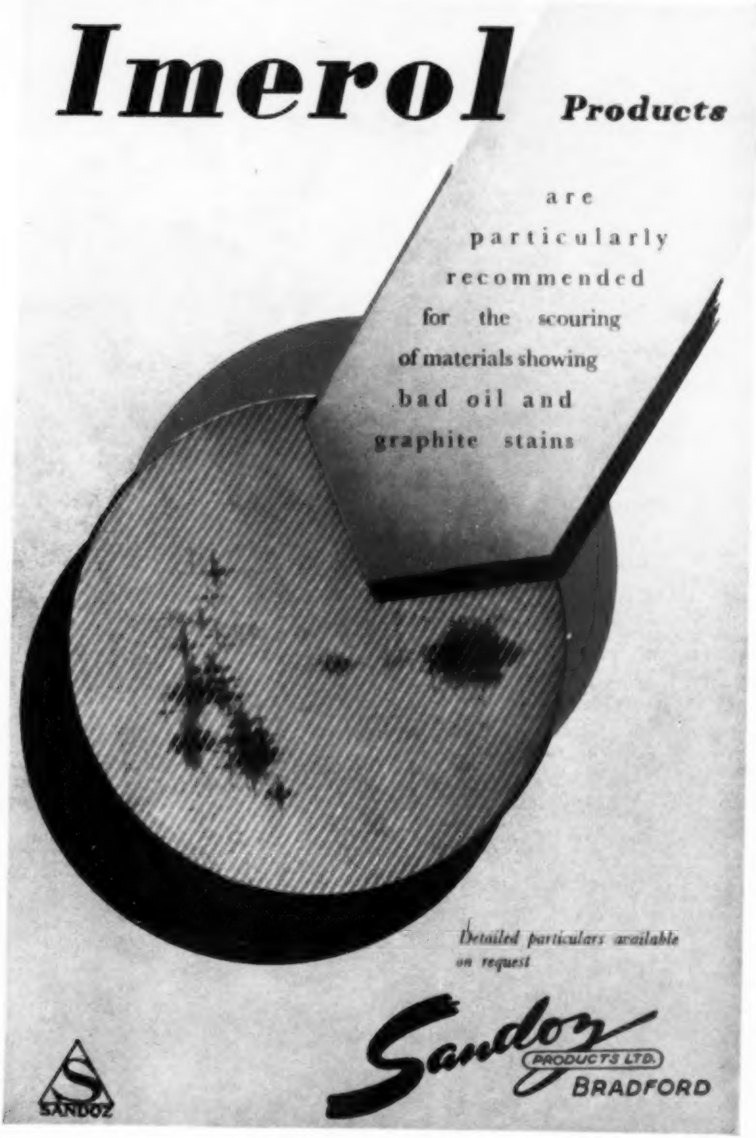
III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Fifty Years of Textile-auxiliary Chemistry. K. Götze. *Melliand Textilber.*, **33**, 51–57 (Jan.); 147–152 (Feb. 1952).

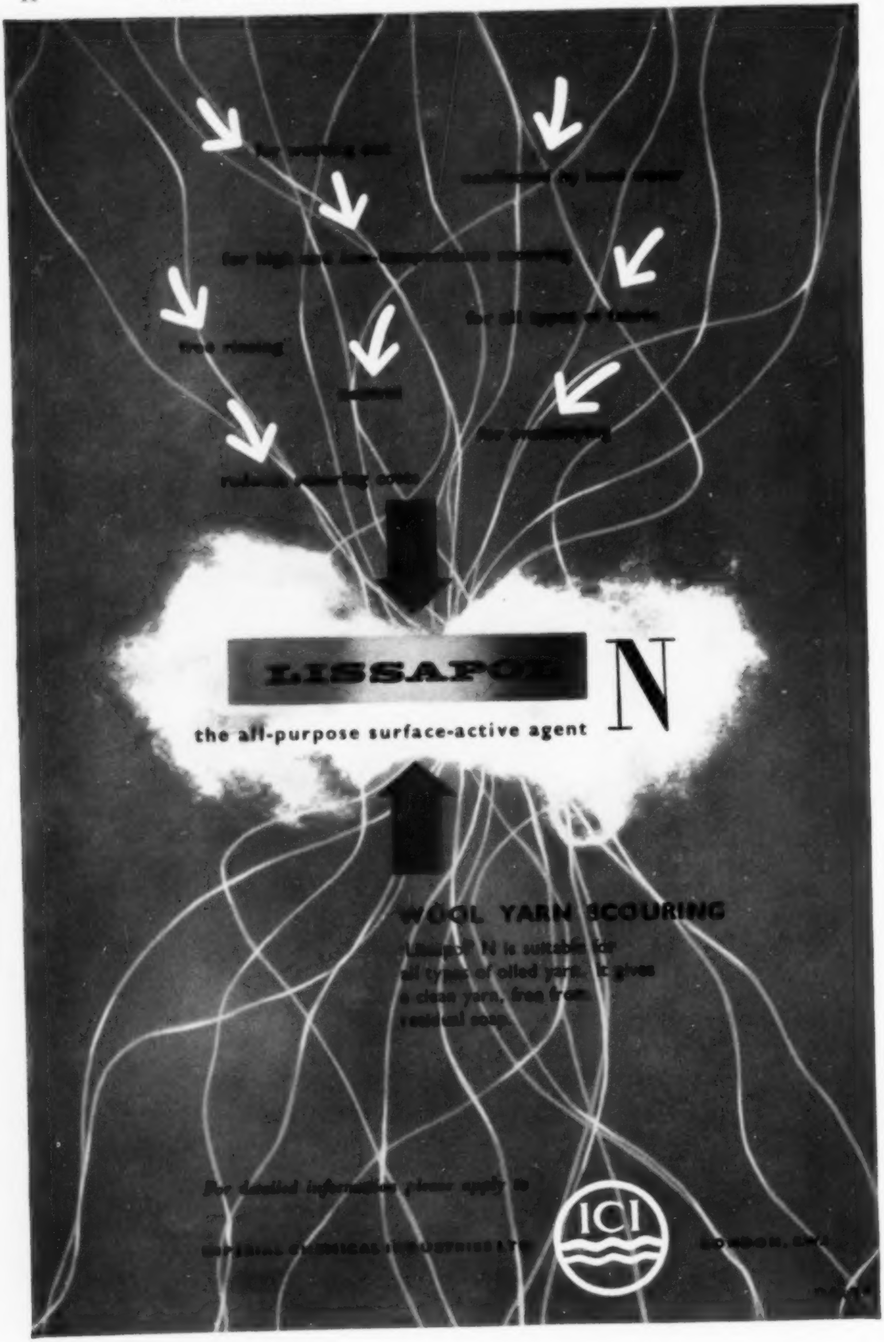
A review is given of surface-active auxiliary products—their nature and properties, and their historical development. There are 48 references. A. E. S.

Industrial Utilisation of Saponin as Textile Auxiliary. I—Wetting and Lime Soap Dispersing Properties. I. S. Uppal and R. L. Mehta. *J. Sci. Industr. Res. India*, **10 B**, (8), 190–195 (1951): *J. Textile Inst.*, **43**, A 102 (Feb. 1952).

The preparation of saponin from the pericarp of soapnut


Imerol Products
are particularly recommended for the scouring of materials showing bad oil and graphite stains
Detailed particulars available on request
Sandoz
PRODUCTS LTD.
BRADFORD
SANDOZ

face centre


LISSAPOL N
the all-purpose surface-active agent
WOOL YARN SCOURING
Lissapol N is suitable for
all types of oiled yarn. It gives
a clean yarn, free from
residual soap.
For detailed information please apply to
ICI

is described. Experiments have been carried out to investigate the wetting power and calcium soap dispersing power of saponin as compared with other detergents. Results are presented, and these show that saponin is superior to the proprietary detergents ordinarily used in the textile industry, particularly in respect of its performance in the presence of hard waters, at high temperatures, and in neutral or alkaline media. C. J. W. H.

Action of Disubstituted Benzene Derivatives on Soap Coacervates. H. L. Booij. *Rec. Trav. chim.*, **71**, 101–107 (Jan. 1952).

The effect of disubstituted benzene derivatives on the formation of Teepol (Shell) and oleate coacervates is measured as the change in concn. (presumably of added salt) required to produce coacervation. With benzene derivatives containing a hydrophilic "anchor" group and a hydrophobic second substituent (e.g. cresols) the activity is in the order $p > m > o$. Where the second substituent is chlorine the sequence is sometimes reversed (e.g. chloroanilines). This also occurs when the "anchor" group is electron-donating and the second substituent electron-attracting (e.g. nitrophenols), and may be due to the second substituent becoming relatively hydrophilic. The location of added substances in the coacervate micelle is mentioned. A. J.

Non-Newtonian Flow of Dilute Polymer Solutions. III—Sodium Carboxymethyl Cellulose in Solutions of Sodium Chloride. F. Akkerman, D. T. F. Pals, and J. J. Hermans. *Rec. Trav. chim.*, **71**, 56–63 (Jan. 1952).

The intrinsic viscosity of very dilute solutions of carboxymethyl cellulose in NaCl soln. is measured using a modification of the method of de Wind and Hermans (*ibid.*, **70**, 521, 615 (1951)). NaCl concn. x is related to polymer concn. c by the equation $x + kc = x_0$. Values of x_0 are 0·024, 0·012, 0·003, and 0·0018. The lowest suitable polymer concn. is 0·004 g./100 ml. From the experimental results the effect of shear rate on intrinsic viscosity can be calculated, and is found to be considerably greater than the values calculated from the theory of Kuhn and Kuhn (*Helv. Chim. Acta*, **28**, 97 (1945)); e.g. with a shear rate increase from 0 to 800 sec.$^{-1}$, the calc. decrease in $[\eta]$ = 5%, observed = 15%. The increased effect may be due to electrostatic effects or particle interaction. A. J.

Hydrolysis of Wool Wax and Related Esters of High Molecular Weight. III—Heterogeneous Reaction: Water-in-oil Emulsions. J. L. Horner and E. V. Truter. *J. Appl. Chem.*, **1**, (Suppl. Issue 2), S 81–S 85 (1951).

Wool wax can be saponified easily with aqueous alkali in the form of a water-in-oil emulsion. Under favourable conditions complete hydrolysis may be achieved within 1 hr. The most convenient catalysts are the products of the reaction, which should be added to the wax before emulsification. Salt, added to the aqueous phase, also catalyses the hydrolysis. The general method of saponification as a water-in-oil emulsion is applicable to any water-insoluble ester. Cholesterol and other higher alcohols are good stabilisers of water-in-oil emulsions, but they tend to inhibit the reaction, for they occupy the interface and prevent the reactants from coming into contact. When used, their concentration should be restricted to the minimum required for emulsion stability. Soaps, when salted out, are not as good stabilisers as the higher alcohols, but they do not block the interface and are thus better catalysts. W. K. R.

PATENTS

Textile-lubricating Compositions. Standard Oil Development Co. *BP* 668,292

Light mineral oil mixed with 5–20% by weight of an oil-soluble mahogany sulphonate and 0·5–3·0% of a polyoxyethylene derivative of a fatty acid partial ester is an efficient fibre lubricant and is readily removed by normal scouring. C. O. C.

Solubilising Agents for Monoalkyl Sulphosuccinates. American Cyanamid Co. *USP* 2,567,159

Water-soluble chromium salts, e.g. Cr sulphate, chloride, or nitrate, are excellent solubilising agents for the higher monoalkyl sulphosuccinate salts, even in presence of sodium sulphate and other salts which normally precipitate the sulphosuccinates. C. O. C.

Polyether Derivatives of Acetic Acid and their Salts—Detergent and Wetting Agents. S. *BP* 669,936

Polyether derivatives of acetic acid, or their salts, useful as detergents, wetting agents, etc., are obtained by treating a compound of formula $R\cdot[O\cdot A]_n\cdot OH$ (R = Alk or Ar; A = alkylene of 2–4 C; n = a whole number) with a monohalogenoacetic acid or a salt thereof and, if the free acid is desired, acidifying the reaction product. C. O. C.

Detergents. Monsanto. *USP* 2,572,805

Ethylene oxide–abietic acid condensates used in combination with ethylene oxide–mercaptan condensates form detergents which do not tend to form "grease-balls". C. O. C.

Sulphonated Arylalkane Detergents. California Research Corpn. *BP* 669,312–3

Production is described. C. O. C.

Sulphonation of Alkyl Aryl Hydrocarbons—Detergents. Lever Brothers & Unilever. *BP* 669,899

Use of SO_3 vapour in an inert gas as the sulphonating agent gives a 95% yield with almost complete freedom from side reactions. C. O. C.

Improving the Detergency of Water-soluble Salts of Alkyl Sulphates. Shell Development Co. *USP* 2,567,645

The detergent properties of alkyl sulphates of 10–25 C are improved by mixing them with a soap, the best effect being obtained by forming the soap in an aqueous solution of the alkyl sulphates. C. O. C.

Reducing the Corrosive Action of Aqueous Solutions of Surface-active Alkyl Sulphate Salts or Alkyl- or Alkaryl-sulphonates. N.V. De Bataafsche Petroleum M.-S. *BP* 669,081

Addition of not > 10% by weight on the surface-active substance, and not > 5% by weight on the weight of the solution, of a water-soluble silicate, chromate, or borate effectively represses the corrosive action of the solution. C. O. C.

Stabilising Tetrachloroethylene. Diamond Alkali Co. *USP* 2,567,621

Compounds of formula $R^1R^2CX\cdot NO_2$ (R^1 = H, CH_3, or C_2H_5; R^2 = H or CH_3 but only H when R^1 = C_2H_5; X = H or Cl, being only H when R^1 and R^2 = H) stabilise commercial tetrachloroethylene to oxidation on exposure to air, light, heat, moisture, and metals. C. O. C.

Superpolyoxyethylene Glycol Esters. Union Carbide & Carbon Corpn. *USP* 2,567,076

The products obtained by treating a polyoxyethylene glycol of average mol. wt. 4000–12,000 with not less than half its molecular equivalent of an acyl halide of a dicarboxylic acid are wax-like, water-soluble, and suitable as components for coating and impregnating compositions, water paints, and textile sizes. C. O. C.

2-Pyrrolidone and its *N*-Alkyl, *N*-Hydroxyalkyl, and *N*-Acyl Derivatives—Textile and Dyeing Assistants. Basf. *BP* 668,961

2-Pyrrolidone and its *N*-alkyl, *N*-hydroxyalkyl, and *N*-acyl derivatives increase the speed of dissolution of mercerising and wetting agents and improve the stability of their solutions; they can be used as solvents instead of alcohol for the coupling components of azoic dyes; and are levelling and penetrating agents for use with sulphur, vat, and acid dyes. C. O. C.

Water-insensitive Titanated Starch. DuP. *USP* 2,570,499

Starch is readily rendered insensitive to water by treating it with a titanium compound. Thus a starch paste containing hydrated precipitated TiO_2 used as an adhesive becomes insoluble in water under the influence of heat and pressure. C. O. C.

Warp Size. Monsanto. *USP* 2,570,830

A solution of 138 gal.(U.S.A.) water, 140 lb. maize starch, 11 lb. tallow, and 6 gal.(U.S.A.) of 15% aqueous colloidal silica is an excellent size for warps. C. O. C.

Size. ICI. *BP* 669,089

An aqueous solution of a cellulose ether, e.g. a carboxyalkyl cellulose, and a polymeric unsaturated aliphatic carboxylic acid, e.g. polymethacrylic acid, not only is an excellent size for yarns but also tends to prevent their collecting dirt and assists in subsequent scouring. C. O. C.

Fluid-stable Starch Adhesives and Sizes. American Cyanamid Co. *BP* 669,201

Aqueous dispersions of converted starch (10–60% starch content) are rendered fluid-stable by addition of 0·1–30·0%, on the dry weight of the starch, of a lower alkyl- or lower hydroxyalkyl-dicyandiamide.

BP 669,202

A similar result is obtained by using an ammonium, alkali metal, alkaline earth metal, or Mg salt of dicyanimide CN·NH·CN.

BP 669,203

0·5–25%, on the dry weight of the starch of dicyanoguanidine or a lower alkyl-dicyanodiguanidine is used. C. O. C.

Hydroxyalkylamine and Morpholine Salts of Polymerised Olefin Sulphonic Acids—Sizes and Coating Compositions. DuP. *USP* 2,570,094

Polythenes and related olefinic polymers are treated with gaseous Cl_2 and SO_2 while irradiated by actinic light. The resulting polysulphonyl derivatives are treated with oxyamine compounds, e.g. polyalkanolamines or the morpholines, in presence of water to hydrolyse the SO_2Cl groups and simultaneously convert the resulting sulphonic acids into the corresponding oxyamine salts. The solubility of the products depends upon the S content of the polysulphonyl compound. If the S content is 4–7%, then the completely neutralised product is water-soluble and suitable as size for warps. If the S content is < 4%, then the product is only sparingly soluble in water but may be thinned for working purposes with an alcohol-water mixture and can be used where a water-resistant size or coating is desired. C. O. C.

Nitrogenous Thermosetting Resins. Röhm & Haas Co. *BP* 667,888

Formaldehyde (2–3 mol.), urea (1), and oxazolidine (0·02–0·5) are heated together at pH 1·5–10·0, preferably 4–7. The products are much more soluble than formaldehyde–urea condensates and are particularly suitable for treating textiles and paper. C. O. C.

Organopolysiloxanes—Hydrophobic Agents and Paint and Varnish Vehicles. Corning Glass Works. *USP* 2,567,110

Organosiloxanes are prepared by treating an alkali metal salt of an organosilanol with a silicon halide. C. O. C.

Water-repellent Agents. Minnesota Mining & Manufacturing Co. *BP* 669,030

Textiles can be rendered water-repellent by treatment with an organic compound of Si containing at least one non-hydrolysable tertiary carbinoxy (alkoxy, aryloxy, or heterocyclic-oxy) group and at least one readily hydrolysable N-containing group attached to the Si atom, each readily hydrolysable group forming a non-acidic product when hydrolysed. C. O. C.

Water-dispersible Pastes of Gas Fading Inhibitors for Acetate Rayon Dyes. DuP. *USP* 2,567,130

Stable pastes of *NN'*-diphenylethylenediamine and related gas fading inhibitors are obtained by dispersing them in an aqueous mixture of either polyvinyl alcohol or methyl cellulose to which an oil-like high-boiling non-volatile water-immiscible liquid, e.g. dimethyl phthalate, has been added, the dispersion being finally passed through a homogeniser. C. O. C.

Fabric-finishing Composition. Perfex Co. *USP* 2,567,722

A laundry finishing composition that can be applied to dark-coloured fabrics or silk or artificial fibres consists of a water-soluble carbohydrate, e.g. starch or methyl cellulose, a wax, and preferably a wetting agent and a stabiliser, e.g. gum arabic. C. O. C.

Rotproofing Agent. Hercules Powder Co. *BP* 669,823

Stabilised rosin amines or their "co-ordinate covalent salts", e.g. dehydrogenated rosin amine or its co-ordinate covalent complex copper salt, are efficient rotproofing agents. C. O. C.

Insect Repellent. U.S. Secretary of the Army. *USP* 2,567,199

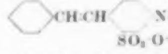
Fabric impregnated with *cyclo*hexyl phenyl ketone remains repellent to *Aedes ægypti* for over ten days.

USP 2,567,200

The dimethyl ether of pyrogallol confers repellency for over five days. C. O. C.

Polyarylethylenearylsulphonic Esters—Intermediates for Optical Bleaching Agents. Gy. *BP* 668,199

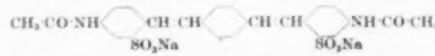
Aryl *p*-nitrotoluene-*o*-sulphonates are condensed with aromatic aldehydes to give stilbene derivatives useful, among other things as intermediates for making optical brighteners. Thus the compound—



is prepared by heating phenyl *p*-nitrotoluene-*o*-sulphonate with benzaldehyde and a little piperidine at 135–145°C. for 1 hr. R. K. F.

1:4-Di(acylaminostyryl)benzenedisulphonic Acids—Optical Bleaching Agents. Gy. *BP* 668,200

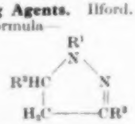
The aryl 4':4''(or 2':2'')-dinitro-1:4-distyrylbenzene-2':2''(or 4':4'')-disulphonates of *BP* 668,199 (above) are reduced, hydrolysed, and acylated to the corresponding bisacylamino-disulphonic acids, which are blue-fluorescing optical brighteners with affinity for animal and vegetable fibres. Thus the compound—



is made by first reducing the corresponding dinitrodisulphonic ester with iron and acetic acid, hydrolysing with alcoholic NaOH, and finally diacetylating in alkaline aq. soln. with acetic anhydride. R. K. F.

Optical Bleaching Agents. Ilford. *BP* 669,590

Compounds of formula—



(R^1 = subst. or unsubst. aromatic hydrocarbon residue containing no nitro group; R^2 = subst. or unsubst. aromatic hydrocarbon residue free from sulphonic, carboxylic, nitro, or cyano groups; R^3 = H, a subst. or unsubst. hydrocarbon group containing no nitro group, or furyl), e.g. 1:3-diphenylpyrazoline or 3-phenyl-1-*p*-sulphophenylpyrazoline, have strong bluish fluorescence in daylight, which is reduced slightly in artificial light. Those of them that are colourless, white, or pale yellow may be used as optical bleaching agents.

BP 669,591

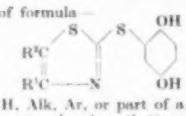
1:3-Diarylpyrazolines which may contain a 5-substituent are obtained by treating a 1-arylpyrazoline or its 5-substituent in basic medium with a diazotised primary arylamine. C. O. C.

Polyvinyl Butyral Organosols—Coating Compositions. Shawinigan Resins Corpn. *USP* 2,571,992

A mixture of finely divided polyvinyl butyral with ethylene glycol is a useful coating composition for application by either brush or spreading techniques. C. O. C.

2-(2-Thiazolylthio)-1:4-dihydroxybenzenes—Rubber Antioxidants. United States Rubber Co. *BP* 668,952

Compounds of formula—



(R^1 and R^2 = H, Alk, Ar, or part of a ring system) are antioxidants for natural and synthetic rubber. C. O. C.

Stable Aqueous Dispersions of Vinyl Chloride Polymers and Copolymers. ICI. *BP* 669,778

An aqueous dispersion of 20–60% by weight of a polymer or copolymer of vinyl chloride is rendered mechanically and electrolytically stable by incorporation of up to 5% on the weight of the polymer of a water-soluble salt of a polybenzylsulphonic acid. C. O. C.

Dyes and Textile Assistants (IV p. 219).
Polyvinyl Chloride Compositions resistant to Fungi and Bacteria (XIII p. 232).
Film-forming Polyvinyl Acetate Emulsions (XIII p. 233).

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

I.G. Farbenindustrie, Hoechst a. M.—Dyes and Textile Assistants. FD 513/51* (PB 70,055–6, 70,150, 70,175–6, 70,180, 70,183, 70,222, 70,249–50, 70,253–7, 70,358–9, 70,361, 70,407, 73,377, 73,485, 73,497, 73,632, 73,849, 74,123, and 74,152).

A very large number of reports on the manufacture and application of dyes and textile assistants, written during 1930–46 and contained in 26 microfilms. C. O. C.

*Photocopies available from T.I.D.U., D.S.I.R., Cunard Building, 15 Regent Street, London S.W.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.—see J.S.D.C., 66, 53 (Jan. 1950).

p-Nitroaniline by Hydrolysis of 4:4′-Dinitrodiphenylurea. I. M. Kogan and D. F. Kutepov. *J. Gen. Chem. U.S.S.R.*, **21**, 2028–2033 (Nov. 1951).

p-Nitroaniline is prepared in good yield by the hydrolysis of 4:4′-dinitrodiphenylurea by means of aq. NH_3 (5 hr. in an autoclave at 125°c.). Hydrolysis by acids is incomplete; hydrolysis by caustic alkali is incomplete and is accompanied by decomposition of the product. A. E. S.

Sulphonation Reaction. XIX—Preparation of 1:3:5-Naphthalenetrisulphonyl Chloride. A. A. Spryskov. *J. Gen. Chem. U.S.S.R.*, **21**, 2022–2027 (Nov. 1951).

The yield of 1:3:5-naphthalenetrisulphonyl chloride obtained by the action of a large excess of chlorosulphonic acid on the 1:5-disulphonyl chloride depends greatly on the quality of the chlorosulphonic acid. Technical acid gives low yields; vacuum-distilled acid, which contains 2·3% of H_2SO_4, gives yields not > 85%; and acid that has been fractionated at atmospheric pressure gives excellent yields of up to 99·7%. The variation in yield is attributed to the unfavourable effect of sulphuric acid and to the favourable effect of pyrosulphuryl chloride $S_2O_5Cl_2$, which is present in acid distilled under atmospheric pressure. A. E. S.

Disulphonation of Carbazole. V. F. Borodkin. *J. Appl. Chem. U.S.S.R.*, **24**, 1202–1207 (Nov. 1951).

Contradictions in the literature are resolved—Sulphonation of carbazole with 2 equiv. of monohydrate leads to two (not three) disulphonic acids (1:6 and 3:6). The use of one equiv. of monohydrate leads to the same products, 50% of the carbazole being recovered unchanged. Sulphonation with excess of 67% H_2SO_4 gives the 3:6 acid and some trisulphonic acid. The acids cannot be characterised by the melting points of the sulphonyl chlorides or sulphamides, as these decompose, on heating, at indefinite temperatures. "Melting points" given for these in the literature are erroneous. A. E. S.

Mechanism of the Reduction of Diazonium Salts—Reduction by Ethanol. A. F. Rekasheva and G. P. Miklukhin. *Doklady Akad. Nauk S.S.S.R.*, **80**, 221–223 (11 Sept. 1951).

Diazonium salts ($R{\cdot}N_2Cl$: R = p-$NO_2{\cdot}C_6H_4$, m-$Cl{\cdot}C_6H_4$, and p-$CH_3(o$-$NO_2)C_6H_3$) are reduced with heavy ethanol C_2H_5OD. The reduction products RH are found to be practically free from deuterium, and their hydrogen must therefore arise from the ethyl group of the ethanol. Since alcohols of the benzyl alcohol type will reduce diazonium salts, it must be supposed that the hydrogen comes in all cases from the α-carbon, e.g.—

$$R{\cdot}N_2Cl + CH_3{\cdot}CHH^{*}OH^{**} = RH^{*} + N_2 + H^{**}Cl + CH_3{\cdot}CHO$$

A. E. S.

Fading Rates of Monoazo Dyes. E. Atherton and R. H. Peters. *J.S.D.C.*, **68**, 64–66 (Feb. 1952).

Some Developments in Dispersed Azo Dyes for Cellulose Acetate Rayon and Nylon. E. Stead and A. Murray. *J.S.D.C.*, **68**, 15–16 (Jan. 1952).

Discoloration of a Dye Liquor by Ultrasonic Vibration. I—An Azo Dye. T. Azami and T. Hinohara. *J. Chem. Soc. Japan, Pure Chem. Sectn.*, **72**, 453–455 (1951): *Chem. Abs.*, **46**, 1843 (10 March 1952).

A 0·003% aq. soln. of Congo Red turned bluish purple when irradiated for 10 min. with ultrasonic waves of 930 kilocycles/sec. This is due to CO_2 being dissolved, so altering the pH from 6·5 to 3·5. If air was excluded the solution became brownish purple. This is attributed to decomposition of the dye at high pH. C. O. C.

Carcinogenic Effect of Some Fat-soluble Azo Dyes. C. Hackmann. *Z. Krebsforsch.*, **57**, 530–541 (1951): *Chem. Abs.*, **46**, 1146 (10 Feb. 1952).

In experiments on feeding rats for approx. 2 yr. on various dyes, the following were shown to have some carcinogenic effect—4-(2-naphthylazo)-3-aminoaniline, 4-(2-hydroxy-1-naphthylazo)-3:3′-dimethylazobenzene, 4-phenylazo-3-methyl-1-phenyl-5-pyrazolone, 4:4′-bis(2-hydroxy-1-naphthylazo)-3:3′-methoxytriphenylmethane, 4-(4-hydroxy-1-naphthylazo)-1-ethoxybenzene, 2-(2-chloro-4-nitro-1-phenylazo)-4-methyl-5-methoxyaniline, 4-(3-methyl-4-hydroxy-1-phenylazo)azobenzene, and 4-p-nitrophenylazodiphenylamine. 4:4′-Bis-(1-hydroxy-4-*iso*hexyl-2-phenylazo)diphenylamine, 4-(2-ethylamino-1-naphthylazo)azobenzene, 1-phenylazo-2-naphthol, 4-naphthylazo-1-naphthylamine (which is quite toxic), 4-(4-amino-1-phenylazo)-1-naphthylamine, and 2-p-acetamidophenylazo-4-methylphenol had no effect. C. O. C.

Colloidal Electrolytes. I—Influence of Radicals on Conductivity of Dye Acids. N. Urabe and M. Sakaguchi. *J. Electrochem. Soc. Japan*, **19**, 224–226 (1951): *Chem. Abs.*, **46**, 1843 (10 March 1952).

The equivalent conductivity and pH titration curves of Direct Black G, Direct Blue 2B, and Acid Blue Black indicate that aggregation of dye ions becomes large as the acidity of the solutions decreases, the number of OH, SO_3H, and NH_2 radicals in the dye molecule increases, and the mol. wt. increases. C. O. C.

Sulphonation of Alicylic Compounds—Action of Chlorosulphonic Acid on *cyclo*Pentanone—Wool Dyes. A. R. Poggi, G. Serchi, and G. Fabiani. *Ann. Chim.* (Rome), **40**, 68–79 (1950): *Chem. Abs.*, **46**, 1458 (25 Feb. 1952).

*cyclo*Pentanone treated with chlorosulphonic acid under four sets of conditions in three cases yielded acidic products of high mol. wt., difficult to purify but having good wool-dyeing properties. In the fourth case *cyclo*pentanone was treated with chlorosulphonic acid in equimolecular proportions at 5°c., 2–3 min. being taken to add the acid to the *cyclo*pentanone; after 5 min. the mixture was removed from the cold bath, stirred for 20 min., washed, and filtered. The black-brown flocculent crystals obtained were dissolved in aq. Na_2CO_3, precipitated with HCl, washed with water, and dried at 100°c. The product, $C_{45}H_{44}SO_{13}$, dyes wool a brilliant, fast, uniform brown. C. O. C.

Triphenylmethane Dyes from Thiodiphenylamine and Phenoxazine. R. Wizinger and S. Chatterjee. *Helv. Chim. Acta*, **35**, 316–329 (Feb. 1952).

The condensation products of benzophenone, methoxybenzophenones, coumarin, and flavone with thiodiphenylamine give a red coloration with alcoholic alkali; on acidification the original greenish colour returns. These observations stimulated research on the absorption spectra of these and related compounds. Ring closure by S and O atoms was found to result in a strong bathochromic change. An attempt is made to interpret the results in terms of modern physical organic chemistry. G. L.

Triarylmethane Dyes containing a Thiophen Ring. C. D. Mason and F. F. Nord. *J. Org. Chem.*, **16**, 722–727 (1951): *Chem. Abs.*, **46**, 1537 (25 Feb. 1952).

An account of the preparation of thiophen dyes and their triarylmethane-type derivatives together with an interpretation of the bathochromic effect of thiophen in triarylmethane dyes. C. O. C.

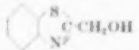
Dyes of the Di- and Tri-pyridylmethane Series. IV—Monopyridyl Analogues of Derivatives of Diphenylmethane. V. M. Berezovsky. *J. Gen. Chem. U.S.S.R.*, **21**, 1903–1909 (Oct. 1951).

Previous work has shown that pyridyl analogues of triphenylmethane dyes and of related substances are coloured only when three *para* auxochromic groups are present. Thus, the analogues of the salts of Malachite Green and of Michler's hydrol or ketone are colourless. It is now shown that when only *one* p-dimethylaminophenyl of Michler's ketone or hydrol, or of the corresponding thioketone, is replaced by the analogous dimethylamino-β-pyridyl radical, the resulting compounds are coloured in acid solution. The colour relations observed are tabulated, and the syntheses of the new dyes described. A. E. S.

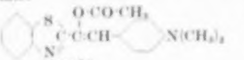
Synthesis of Oxazolotrimethincyanine and its Trinuclear Dyes. S. Taki. *Repts. Sci. Research Inst. (Japan)*, **25**, 224–226 (1949).

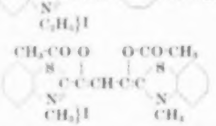
2-Hydroxymethylbenzothiazole and its Reactions. V. M. Zubarovsky. *J. Gen. Chem. U.S.S.R.*, **21**, 2055–2064 (Nov. 1951).

2-Hydroxymethylbenzothiazole—


(I)

which has not been described previously, is prepared in good yield by the condensation of glycollic acid with o-aminothiophenol. The OH of I has "phenolic" properties, and a cryst. Na salt can be obtained from aq. alkaline soln. 2-Bromomethylbenzothiazole, obtained from I by the action of HBr, is very reactive (e.g. to Na_2S, NH_3, amines, alkoxides, thiols, etc.), the Br being readily replaced by a great variety of radicals. Some of these products, and the esters and ethers of I, are considered for use as intermediates in the preparation of cyanine dyes. Their application, however, is not always straightforward, for their quaternary salts are often converted on heating into the quaternary salt of 2-methylbenzothiazole. A new dimethin styryl dye (II, absorption max. in ethanol 498 mμ.) is obtained from a quaternary salt of the acetyl deriv. of I and p-dimethylaminobenzaldehyde, and a new trimethin thiacarbocyanine (III, absorption max. in ethanol 554 mμ.) from the same acetyl cpd. and ethyl orthoformate.


(II)


(III)

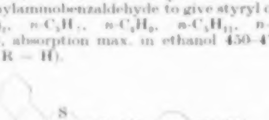
A. E. S.

Condensation of o-Aminothiophenol with α-Hydroxy Acids. V. M. Zubarovsky. *J. Gen. Chem. U.S.S.R.*, **21**, 2199–2205 (Dec. 1951). **Alcohols and Ketones of the Benzothiazole Series.** V. M. Zubarovsky and M. D. Rikelman. *Ibid.*, 2205–2210.

In the reaction described in the previous abstract for the preparation of 2-hydroxymethylbenzothiazole, glycollic acid is replaced by other hydroxy acids. Monobasic, e.g. lactic and mandelic, acids give analogous products; seven of these and their corresponding ketones are described. Malic and tartaric acids give mixtures in which the hydroxy cpd. is a minor constituent. A. E. S.

Synthesis of 2-Alkyl-α-naphthothiazoles II. V. M. Zubarovsky and S. N. Fidel. *J. Gen. Chem. U.S.S.R.*, **21**, 2064–2068 (Nov. 1951).

Previous work (*Chem. Abs.*, **42**, 906*b* (10 Feb. 1948)) on the synthesis of 2-alkyl-α-naphthothiazoles is extended, eight further homologues being prepared. Quaternary salts of six of the new compounds are treated with p-dimethylaminobenzaldehyde to give styryl dyes (I: R = $CH(CH_3)_2$, n-C_3H_7, n-C_4H_9, n-C_5H_{11}, n-C_6H_{13}, and n-C_7H_{15}), absorption max. in ethanol 450–473 mμ. (537 mμ. for R = H).


(I)

A. E. S.

Cyanine Dyes. VI—Properties of 7:7′-Bisdimethylaminothiacarbocyanines. I. I. Levkoev and B. S. Portnaya. *J. Gen. Chem. U.S.S.R.*, **21**, 2050–2055 (Nov. 1951).

Oxidation of m-thioacetamidodimethylaniline with potassium ferricyanide in alkaline soln. yields 7-dimethylamino-2-methylbenzothiazole. The methiodide or alkyl p-toluenesulphonate of this condenses in pyridine soln. with an ortho-ester of a carboxylic acid to give a 7:7′-bisdimethylaminothiacarbocyanine, either unsubstituted (using orthoformic ester) or substituted in the 9-position with methyl or ethyl (using orthoacetic or orthopropionic ester respectively). The absorption spectra of the new dyes are compared with those of the already known isomers having dimethylamino groups in the 5:5′- and 6:6′-positions. The results are analogous to those previously found for dihydroxy- and dimethoxy-thiacarbocyanines—the introduction into thiacarbocyanines of dimethylamino groups in the 7- and 7′-positions displaces the absorption max. (540–560 mμ.) towards the longer waves by only 5–11 mμ., as compared with a displacement of 51–55 mμ. for the 5:5′- and 6:6′-derivatives. A. E. S.

***mero*Cyanine Dyes derived from Rhodanine. II—Properties of Dimethin*mero*cyanines having various Nitrogen-containing Heterocyclic Residues.** M. V. Deychmeyster, Z. P. Sytnik, and E. B. Lifshits. *J. Gen. Chem. U.S.S.R.*, **22**, 166–175 (Jan. 1952).

Fifteen dyes derived from rhodanine—14 dimethin*mero*cyanines and one monomethinoxanine (two rhodanine residues)—and the 15 corresponding dyes from 3-ethylrhodanine are synthesised and characterised. The second heterocyclic residue of the *mero*cyanine is varied. The absorption max. of each *mero*cyanine is compared with those of the corresponding "symmetrical" cyanines—the carbocyanines corresponding to the second heterocyclic residue—and the synthesised oxanine. It is displaced to the shorter waves, compared with the arithmetical mean of these two values, to an extent that diminishes as the second heterocyclic residue becomes more basic. All the synthesised *mero*cyanines are sensitisers for silver halide emulsions, the most effective being those with thiazole, thiazoline, and α-pyridine residues. A. E. S.

Bisulphite Compounds. XIV—Bisulphite Compound of 2-Methyl-1:4-naphthaquinone and its Reaction with Phenylhydrazine. V. N. Ufimtsev and M. I. Chernyak. *J. Gen. Chem. U.S.S.R.*, **21**, 1883–1887 (Oct. 1951).

In 2-methyl-1:4-naphthaquinone (I), steric effects prevent the 1-carbonyl group from reacting with reagents such as bisulphite and phenylhydrazine. The bisulphite cpd. of I reacts readily with phenylhydrazine, and this has been taken as evidence that the reaction with bisulphite occurs not at the 4-carbonyl, but in the 3-position, leaving the carbonyl free for reaction with a hydrazine. It is shown, however, that potassium bisulphite reacts with I with the formation of $>C(OH)\cdot SO_3K$ at the 4-position, and that the subsequent condensation of phenylhydrazine occurs also in the 4-position, with elimination of the OH. A. E. S.

3- and 4-Sulphonic Acids of 1:2-Naphtho-(3′:4′)-furazan. S. V. Bogdanov and B. I. Karavaev. *J. Gen. Chem. U.S.S.R.*, **21**, 1915–1918 (Oct. 1951).

1:2-Naphtho-(3′:4′)-furazan (the anhydride of 1:2-naphthaquinone dioxime) is monosulphonated when treated with conc. H_2SO_4 at 160°c. or with oleum at ordinary temp. The product must be the 3- or 4-sulphonic acid, for it yields phthalic acid on oxidation. However, the 4-sulphonic acid remains unchanged when boiled with 60–70% H_2SO_4, whereas the product of direct sulphonation is hydrolysed under these conditions, and must be the 3-acid. The 4-sulphonic acid is made by treating 1-nitroso-2-naphthol-4-sulphonic acid (Na salt) with hydroxylamine hydrochloride in presence of an alkaline neutralising agent, and then boiling the resulting dioxime in alkaline soln.; in absence of a neutralising agent, the product is 2-nitroso-1-naphthol-4-sulphonic acid. A. E. S.

Hydrocarbons formed by Distillation of Benzanthrone with Zinc Dust. III—Violanthrene and *iso*Violanthrene. A. Zinke and R. Ott. *Sitzungsber. Österr. Akad. Wiss., Abt. IIb*, **160**, (6), 723–726 (1951).

The authors have recently shown that distillation of benzanthrone with Zn dust yields small quantities of violanthrene and traces of *iso*violanthrene (*Monatsh. Chem.*, **81**, 1137 (1950)). The small yet quite distinct differences in properties of the two isomers are shown in tabular form. Structural relationships with benzanthrene or perylene and nomenclature are critically discussed. G. L.

Properties of Some Indanthrone and Flavanthrone Derivatives. W. Bradley, E. Leete, and H. E. Nürsten. J.S.D.C., **68**, 116–118 (April 1952).

Structure of Some Indigo Yellows. I—X-Ray, Crystallographic, and Optical Investigations. S. Hotz. *Schweiz. mineralog. petrogr. Mitt.*, **31**, 188–292 (1951): *Chem. Abs.*, **46**, 1838 (10 March 1952).

Studies of Höchst Yellow U, Indigo Yellow 3G (Ciba), and related compounds. C. O. C.

Photochemical Properties of Iron Complexes of Chlorophyll. M. S. Ashkinazi and B. Ya. Dain. *Doklady Akad. Nauk S.S.S.R.*, **80**, 385–388 (21 Sept. 1951).

The iron complex of chlorophyll is photoreduced in soln. by visible light in complete absence of oxygen. The product is very readily re-oxidised. The reactions are followed by changes in the absorption spectra. A. E. S.

Photochemistry of Chlorophyll at the Temperature of Liquid Air. A. A. Kachan and B. Ya. Dain. *Doklady Akad. Nauk S.S.S.R.*, **80**, 619–622 (1 Oct. 1951).

Chlorophyll in alcoholic soln. has a high photochemical stability at liquid-air temp.; in alcohol–ether it fades rapidly, but recovers its original condition on allowing the soln. to attain room temp. The photoreaction is regarded as the liberation of an electron with formation of a positive chlorophyll ion. In alcoholic soln. the reaction is readily reversible, but in presence of ether the positive ion loses a proton to the ether molecule, and the resulting system is metastable at low temp. The results of various authors on the fading of chlorophyll by visible light in oxygen-free soln. are to be explained by the reaction of excited molecules with the solvent. A. E. S.

Pigments and Antibiotic Substances of Fungi and Bacteria. C. Sannié. *Exposés annuels Biochim. méd.*, **6**, 225–280 (1946): *Chem. Abs.*, **46**, 1095 (10 Feb. 1952).

A review with numerous references. C. O. C.

PATENTS

Resorcinolmonosulphonamides—Azo Dye Coupling Components. General Aniline. *BP* 667,772

Resorcinol-4(and 5)-sulphonamides, in which the N atom may carry Alk, Ar, etc. substituents, are azo-dye coupling components, particularly suitable for diazotype reproduction processes because of the high opacity of the dyes they produce. They are prepared from resorcinol-monosulphonic acids by reaction with benzenesulphonyl chloride to acylate the OH groups, followed by treatment with $POCl_3$ or PCl_5 and then with ammonia or an amine. Saponification of the benzenesulphonyl group with NaOH then yields the resorcinolsulphonamide. Thus ammonium 2:4-dihydroxybenzenesulphonate is treated with benzene-sulphonyl chloride in water at 55–65°c. in presence of soda ash, and the diacylated product collected and dried. It is then treated with $POCl_3$, and the sulphonyl chloride so produced is dissolved in benzene and treated with aniline at room temperature. The product is refluxed with dil. aq. NaOH to yield 2:4-dihydroxybenzenesulphonanilide. E. S.

4-Nitro-1-naphthylamine-5-sulphonic Acid. Ciba. *BP* 669,220

Improved yields of 4-nitro-1-naphthylamine-5-sulphonic acid are obtained by conducting the nitration of 1-naphthylamine-5-sulphonic acid at < 0°c., and preferably at -10 to -2°c. E. S.

Azo Compounds from Nitro Compounds. DuP. *USP* 2,551,003

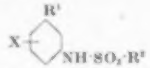
Aromatic nitro compounds, e.g. nitrobenzene, *p*-nitrobenzoic acid, β-nitronaphthalene, and *p*-nitrodiphenyl, are reduced to azo compounds by heating under pressure with alkali-metal hydroxides and a primary aliphatic monohydric alcohol of < 4 C. Thus, all parts being by weight, NaOH (22·5), methanol (72), and nitrobenzene (60) are charged into an autoclave and heated to ca. 100°c., when the internal temperature rises rapidly to 135–150°c. and the pressure to 75–100 lb./sq. in. The reaction is completed by heating to 160°c. for 3 hr. The yield of azobenzene (38–40) is 85–90% of theory. E. S.

2-Hydroxy-3-naphthoic Acid—Preparation of Anhydrous Sodium β-Naphthoxide. DuP. *USP* 2,544,881

Molten anhydrous sodium β-naphthoxide can be made by heating β-naphthol with aq. NaOH in an inert atmosphere to 307–370°c. and reducing the pressure below the equilibrium vapour pressure (172 mm. Hg at 307°c.): at this temperature dehydration occurs rapidly. The molten product, after cooling and grinding in absence of oxygen and water, is ready for conversion into 2-hydroxy-3-naphthoic acid. Thus, all parts being by weight, β-naphthol (242), NaOH (68), and water (140) were mixed in an atmosphere of nitrogen and heated to 308°c. in 1 hr. without agitation. The pressure was reduced to < 170 mm. Hg, and dehydration completed in 10 min. at 324°c. A similarly sized charge heated under vacuum at the more commonly used temperature of ca. 235°c. took several hours to dehydrate completely. E. S.

Colour Formers yielding Non-migratory Azine Dyes. General Aniline. *USP* 2,569,903

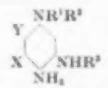
Compounds of formula—



(R^1 = $-NH_2$, $-NHAlk$, $-NAlk_2$, or OH; X = $R^2·SO_2·NH-$, $R^2·SO_2·NH·C_6H_4-(p)$, or $R^2·SO_2·NH·C_6H_4·CH_2-(p)$; R^2 = Alk of > 9 C or Ar which is substantive or contains Alk of > 9 C, R^2 preferably being Ar when R^1 = OH) are colour formers which together with the azine dyes they yield are non-migratory from photographic emulsions. C. O. C.

Colour Developers for Azine Dyes. General Aniline. *USP* 2,570,116

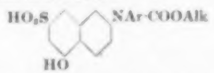
Compounds of formula—



(R^1 = H or Alk; R^2 = Alk, carboxyalkyl, hydroxyalkyl, Ar, carboxyalkylaryl, carboxyalkoxyaryl, hydroxyalkylaryl, or hydroxyalkoxyaryl; R^3 = Alk subst. by a hydrophilic group; X and Y = H, Alk, sulpho, or carboxy or together = atoms necessary to complete a six-membered isocyclic ring) yield yellow dye images when a silver halide emulsion is developed in presence of an open-chain ketomethylene compound, as well as the usual magenta and cyan azine dyes with suitable components. C. O. C.

Urethans of Aryl-J Acids. General Aniline. *USP* 2,542,596

N-Aryl-J acids react readily with alkyl chloroformates to yield urethan derivatives—



which are more stable than the *N*-aryl-J acids, and yield brighter dyes when used as azo coupling components. Thus, 81% phenyl-J acid (37) is dissolved in water (200) containing 6 N-NaOH (36), and ethyl chloroformate (12) added, a temp. of 5°c. being maintained by external cooling. After 15 min. more 6 N-NaOH (17) and ethyl chloroformate (12) are added, and after a further 15 min. a third addition of the reagents, like the second, is made. The ethyl urethan of phenyl-J acid separates as an oil, which slowly crystallises, and may be purified by dissolution in water and salting out.

USP 2,542,597

Alkyl urethans of β-naphthyl-J acid, prepared by reaction of the latter with alkyl chloroformates, are intermediates for the production of azo dyes of good brightness. Bromination of the alkyl urethans of β-naphthyl-J acid in presence of acetic acid and acetic anhydride is also described. E. S.

Furoylaminonaphthols. General Aniline. *USP* 2,543,745

2-Furoyl chloride reacts with aminonaphthols in presence of acid-binding agents to give *N*-furoyl derivatives which are azo-dye coupling components and couplers in diazotype processes and colour photography. Thus, all parts being by weight, 2-furoyl chloride (106) is added dropwise to a stirred suspension of 1-amino-7-naphthol (105) in water (500) containing Na_2CO_3 (100) at 35°c. The sticky precipitate solidifies on standing, and is purified

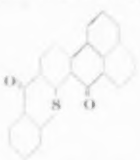
by solution in boiling 10% aq. Na_2CO_3 (1500), reprecipitation with dil. HCl after cooling, and washing with 5% aq. $NaHCO_3$ (1000) at 70°c. E. S.

Monobenzoylated 1:4-Diaminoanthraquinones. Ciba. *BP* 668,303

1-Amino-4-benzoylaminoanthraquinones are made by first oxidising the *leuco*-1:4-diaminoanthraquinone in a mixture of nitrobenzene and a tertiary aromatic amine, e.g. diethylaniline, at 160°c., and then adding an equimol. quantity of a benzoyl chloride at 60–65°c. R. K. F.

Benzanthrone Acridones and Thioxanthones. General Aniline. *USP* 2,546,138

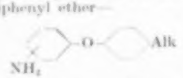
Anthraquinone-2:1-acridones and -2:1-thioxanthones are treated with iron and glycerol in H_2SO_4 to yield the corresponding benzanthrone derivatives, which are intermediates for vat dyes. Thus the compound—



is prepared by heating anthraquinone-2:1-thioxanthone with glycerol and iron borings in 92% H_2SO_4 at 115–120°c. The products are monobrominated by treating with bromine in nitrobenzene containing a little iodine at 160°c. R. K. F.

Monoazo Acid Dyes. Ciba. *BP* 667,590

Acid dyes of good brightness are made by diazotising an *o*- or *p*-aminodiphenyl ether—



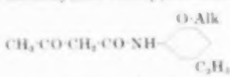
(Alk of 4–9 C; other common substituents, e.g. Hal, may be present), and coupling with an *ortho*-coupling naphtholdisulphonic acid. The aminodiphenyl ethers are made by condensing appropriate *p*-alkylphenols with chloronitrobenzenes, and reducing. Thus, all parts being by weight, 2-amino-4′-*tert.*-amyl-4-chlorodiphenyl ether (14·5) is dissolved in acetic acid (50) and mixed with conc. HCl (19). Sodium nitrite (3·6) as a strong solution is added at < 5°c. The diazo solution is then diluted and added to disodium 1-naphthol-3:6-disulphonate (18) dissolved in water (500) containing soda ash (120) at 0°c. The product dyes wool salmon red from an acetic acid bath. E. S.

Metal-complex Azo Leather Dyes. ICI. *BP* 668,474

The water-soluble metal complexes of nitrosated azo dyes $R^1{\cdot}N{:}N{\cdot}R^2(OH)_2NO$ (R^1 = aryl containing at least one SO_3H; R^2 = aryl of the benzene series in which the OH groups are *meta* to each other, and the NO is *ortho* to an OH) are leather dyes of good light fastness. Thus the monoazo compound naphthionic acid → resorcinol is treated with nitrous acid, and the nitroso compound so obtained heated at 70°c. with $FeCl_3$ under weakly acid conditions, to give a dull brown leather dye. Disazo dyes derived from phloroglucinol, nitrosated and metallised, give similar results. E. S.

Yellow Azo Pigments from Acetoacetarylides. American Cyanamid Co. *USP* 2,542,565

5-Ethyl-2-methoxy(and ethoxy)acetoacetanilides—



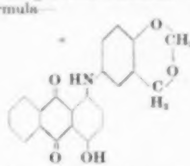
(Alk = CH_3 or C_2H_5) give bright yellow pigments of good solvent resistance by coupling with suitable diazo and tetrazo compounds. Thus 2-nitro-4-ethylphenol is methylated and then reduced to give 5-ethyl-2-methoxyaniline, which reacts with diketen in acetone to yield the *N*-acetoacetyl derivative. On coupling with tetrazotised 3:3′-dichlorobenzidine, a yellow pigment, greener and brighter than the lower homologue given by using 2-methoxy-5-methylacetoacetanilide (acetoacetocresidide), is produced. E. S.

Azo Pigments—Improved Fastness to Bleeding by Treatment with Formaldehyde. DuP. *USP* 2,540,705

By treating azo and azoic pigments with formaldehyde in conc. H_2SO_4, deeper colours, of improved fastness to migration in organic films, are produced. Thus, all parts being by weight, the red pigment 2:5-dichloroaniline → 2-hydroxy-3-naphtho-*o*-toluidide (50) is dissolved in 96% H_2SO_4 (1470), and a solution of paraformaldehyde (5·33) in H_2SO_4 (69·7) is added. After 10–11 min. the solution is drowned in water (6000), and the precipitated maroon pigment filtered off, washed, and dried. E. S.

1-(6-Benzo-1:3-dioxanylamino)-4-hydroxyanthraquinone—Dye for Cellulose Esters. General Aniline. *USP* 2,546,121

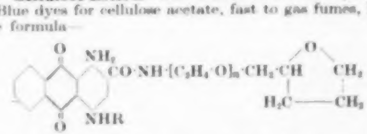
A blue-violet gas-fume resistant dye for cellulose acetate of formula—



is made by refluxing *leuco*-quinizarin with 6-amino-1:3-benzodioxan in ethanol in presence of boric acid and oxidising the product with aq. sodium perborate. R. K. F.

1:4-Diamino-2-β-tetrahydrofurfuryl(poly)oxyethylenecarbamylanthraquinones—Dyes for Cellulose Esters. Eastman Kodak. *USP* 2,549,784

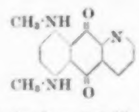
Blue dyes for cellulose acetate, fast to gas fumes, have the formula—



(R = H, Alk, or hydroxyalkyl; n = 1–3). They are made by condensing the corresponding 4-bromo compound with NH_3 or an amine. Thus 1-amino-4-bromoanthraquinone-2-carboxy-β-tetrahydrofurfuryloxyethylamide is refluxed with aqueous methylamine, cuprous iodide, and potassium acetate in pyridine for 3 hr. R. K. F.

α-Aza-anthraquinone Derivatives—Dyes for Cellulose Esters and Superpolyamides. Eastman Kodak. *USP* 2,552,263

leuco-5:8-Dihydroxy-1-aza-anthraquinone is condensed with amines to produce dyes for cellulose esters, which on sulphonation dye wool and silk. Thus the blue dye—



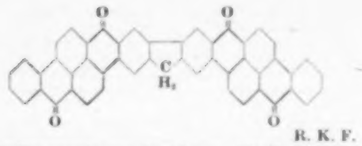
is prepared by refluxing *leuco*-5:8-dihydroxy-1-aza-anthraquinone with methylamine in butanol for 10 hr. and oxidising by passing air into the soln. R. K. F.

Pyranthrone Vat Dyes. Basf. *BP* 668,103

Halogenated pyranthrone derivatives are condensed with 1-aminoanthraquinone 2-aldehyde to give brown vat dyes. Thus, 1-aminoanthraquinone 2-aldehyde is refluxed with dibromopyranthrone, CuO, and Na_2CO_3 in naphthalene. R. K. F.

Benzanthrone Fluorene Derivatives (Bisbenzopyrenequinonofluorenes)—Vat Dyes. General Aniline. *USP* 2,546,139

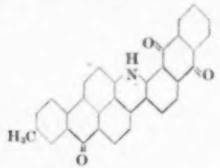
A benzanthrone-3-carboxylic acid chloride is condensed with fluorene, and the resulting compound cyclised by fusing with KOH to give an olive vat dye fast to chlorine. The first stage is carried out by heating the reactants in nitrobenzene at 80°c. in presence of $AlCl_3$. The product is heated at 230°c. in KOH containing one-tenth of its weight of fused sodium acetate to give the dye—



R. K. F.

9-Methylbenzanthrone Acridones—Vat Dyes. General Aniline. *USP* 2,547,044

Benzanthrone-"acridone" dyes of the Indanthren Olive Green B type having CH_3 in position 9 have improved printing properties. They are prepared by condensing 3-bromo-9-chloromethylbenzanthrone with a 1-aminoanthraquinone, after protecting the chloromethyl if necessary, e.g. by converting it to a quaternary ammonium group, and finally cyclising by fusion with alkali. Thus, 3-bromo-9-chloromethylbenzanthrone (obtained by heating 3-bromobenzanthrone with dichlorodimethyl ether in H_2SO_4) is refluxed with 1-aminoanthraquinone in nitrobenzene containing Na_2CO_3 and sodium and copper acetates. The product is cyclised by heating with KOH in ethanol at 119–125°c. for 3·5 hr., when the chlorine atom is also eliminated, to give the dye—



R. K. F.

Halogenomethylated Thioindigoid Dyes. General Aniline. *USP* 2,545,497

Thioindigoid dyes are condensed with e.g. dichlorodimethyl ether in sulphuric acid to produce chloromethyl derivatives, which are converted to water- and spirit-soluble dyes by treating with amines or mercaptans. Thus, Indanthren Brilliant Pink R is stirred at 25–40°c. in sulphuric acid with dichlorodimethyl ether for 19 hr.

R. K. F.

Green Sulphur Dye. DuP. *USP* 2,544,888

A green sulphur dye brighter and yellower than *C.I.* 1006 is produced by treating the *leuco*-indophenol from phenyl-peri acid and *p*-aminophenol with a polysulphide in presence of 2-ethoxyethanol (Cellosolve) and treating the product with aq. alkali-metal cyanide at 75–90°c.

R. K. F.

Phthalocyanines soluble in Hydrocarbons. General Aniline. *USP* 2,547,972

Phthalocyanine derivatives soluble in hydrocarbon solvents and hydrophobic resins, e.g. polystyrene, are made by condensing the chloromethylated phthalocyanines of *USP* 2,435,307 with primary or secondary amines having > 3 C atoms. Thus, copper trischloromethylphthalocyanine is refluxed (135–137°c.) with diisobutylamine, and the product separated by adding ethanol.

R. K. F.

Halogenated Phthalocyanines. Standard Ultramarine Co. *USP* 2,549,842

Green-blue pigments consisting of mixtures of hydrated metal oxides and phthalocyanines with > 8 Hal are prepared by heating together in an inert solvent a halogenated phthalocyanine-forming substance, e.g. tetrachlorophthalic anhydride, urea or a related substance, Cu or one of its compounds, and a halide of Zr, Ti, Sn, Sb, or As. Thus, urea, tetrachlorophthalic anhydride, Cu powder, zirconium tetrachloride, and ammonium perchlorate are mixed, added to trichlorobenzene, and heated at 175–180°c.

R. K. F.

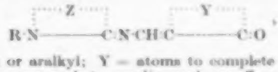
Polymethin Dyes. Kodak. *BP* 669,174

Carbocyanine dyes can be prepared by condensing a *cyclo*ammonium quaternary salt, containing a CH_3 group in α- or γ-position to the quaternary N atom, with a dialkoxymethyl carboxylate, e.g. diethoxymethyl acetate. In some cases neocyanine is formed at the same time as the carbocyanine; and by choice of solvent the reaction can, in some cases, be controlled to yield only carbocyanine and in others to yield only neocyanine, a table of such cases being given.

C. O. C.

Azamerocyanine Dyes. General Aniline. *USP* 2,572,961

Dyes of formula—

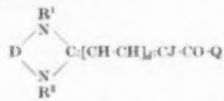


(R = Alk or aralkyl; Y = atoms to complete a 5-membered nitrogenous heterocyclic nucleus; Z = atoms to complete a 5- or 6-membered heterocyclic nucleus) are obtained by refluxing 1 mol. of a 2-amino heterocyclic nitrogenous quaternary salt in presence of a basic condensing agent and a tertiary base with 1 mol. of a 5-membered heterocyclic intermediate having a reactive acetanilido group linked to a C atom of a monomethin chain adjacent to a keto group.

C. O. C.

Photographic Sensitising Dyes. Kodak. *BP* 670,038

Dyes of formula—



(R^1 = Alk, Ar, or alkoxyalkyl; R^2 = subst. or unsubst. Alk or an unsatd. group; D = a bivalent *o*-arylene radical; J = cyano, carbalkoxyl, or acyl; Q = carbalkoxyl or subst. or unsubst. benzofuryl; or J and Q together = non-metallic atoms needed to complete a 5- or 6-membered heterocyclic ring or a 5-membered carbocyclic ring; *d* = 1–3) are of use as photographic sensitisers.

C. O. C.

Pigments for Printing Inks. Sterling Drug. *USP* 2,567,963–5

The pigments obtained by precipitating *C.I.* 640, 671, and 778 with organic ammonium compounds of 14–35 C yield printing inks which are almost completely transparent and thus do not restrict the printer to any definite order of laying down the colours.

C. O. C.

Concentration and Recovery of Carotenoid Pigments from Palm Oil. Colgate-Palmolive-Peet Co. *USP* 2,572,467

Furnace Black. Columbian Carbon Co. *BP* 668,519

The difficulties met with in cooling furnace effluent gases by water sprays are largely eliminated by injecting the water into the hot gases as a mist of particle size 40–80 μ.

C. O. C.

Hydrophilic Carbon Black. Godfrey L. Cabot. *BP* 668,724

Carbon black is rendered readily and spontaneously dispersible in water by exothermal treatment with aq. sodium hypochlorite.

C. O. C.

Carbon Black from High-boiling Aromatic Oils or Tar. Phillips Petroleum Co. *USP* 2,572,734

Carbon Black. Columbian Carbon Co. *BP* 669,511–2

A method of obtaining increased yield when using the process described in *BP* 655,799.

C. O. C.

Carbon Black. Columbian Carbon Co. *BP* 669,968

An improved method of mixing a hydrocarbon with hot furnace gases.

C. O. C.

Calcium Carbonate. ICI. *BP* 668,572

Calcium carbonate of fine particle size and high specific surface area is produced by dehydrating the calcium carbonate hexahydrate obtained by precipitation from aqueous solution in presence of a small amount of glassy sodium polyphosphate of $Na_2O : P_2O_5$ molar ratio 1·12 : 1.

C. O. C.

Calcium Silicate Pigments. R. T. Vanderbilt Co. *USP* 2,573,677

An aqueous calcium chloride–starch complex solution is mixed with aqueous sodium silicate. This procedure permits high rates of production and control of the particle size of the pigment.

C. O. C.

Iron Oxide Pigments. Reymersholms Gamla Industri. *BP* 668,929

Yellow iron oxide mainly composed of submicrocrystalline or cryptocrystalline α-ferric hydroxide (α-FeOOH) can be converted with ferrous hydroxide into

black ferrosoferric oxide, e.g. by boiling the two hydroxides together. Dark green or yellowish-green pigments may be obtained by increasing the yellow hydroxide present beyond the molecular proportion 1 Fe_2O_3 : 1 FeO. C. O. C.

White and Grey Silicon Nitride Pigments. Union Carbide & Carbon Corpn. *USP* 2,572,252

Paints pigmented with white and grey silicon nitride have superior resistance to abrasion and marked chemical inertness. Grey silicon nitride has good opacity and covering power and can be used alone, but white silicon nitride because of its inferior opacity and covering power needs another pigment to be used with it. C. O. C.

Seed for Production of Titanium Dioxide. M. J. Mayer. *USP* 2,571,150

Polyarylethylenearylsulphonic Esters—Intermediates for Optical Bleaching Agents (III p. 218).
Whiting in the Paint Industry (V below).
Phloroglucinol as Developer for Diazotisable Dyes (VIII p. 228).
Photodichroism of Dye-Gelatin Films. I—Relaxation Phenomena. II—Correct Dichroism–Time Curves considering Relaxation Phenomena (IX p. 228).
Magnetic Dichroism of Dye–Gelatin Films—I (IX p. 229).

V—PAINTS; ENAMELS; INKS

Whiting in the Paint Industry. G. E. Bessey and K. A. Lamminan. *Paint*, **22**, 85–90 (March 1952).

An account of the manufacture of whiting from chalk followed by a review of its present and possible future uses in the paint industry. C. O. C.

PATENTS

Flushed Methyl Violet Inks. Sun Chemical Corpn. *USP* 2,567,281

Addition during flushing to the ink vehicle-pigment mixture of a higher fatty acid and a product obtained by treating a fatty acid with a compound of formula—

$$H_2N{\cdot}[CH_2{\cdot}CH_2{\cdot}NH]_n{\cdot}CH_2{\cdot}CH_2X$$

(n = 1–4; X = NH_2 or OH), where the vehicle consists of a neutral liquid containing a resinous binder, results in free-flowing inks which are excellent toners for carbon black inks. C. O. C.

Zein Inks. Time. *USP* 2,570,353

An ink that transfers well from all kinds of metal surfaces without specking consists of a zein-base ink to which 2–10% of a lubricating and wetting agent has been added, e.g. propylene glycol containing 11–20% zein, 15–25% pigment, and 2–10% of an 80 : 20 mixture of light mineral oil and a sulphated vegetable or sulphonated mineral oil. C. O. C.

Films containing Metallic Flake Pigments. DuP. *USP* 2,570,856

Paint or other soft film containing metallic pigments of which not < 50% are flakes and not < 10% ferromagnetic is treated with a magnetic field at 20–75° to the plane of the film, which is kept in the magnetic field until it has dried sufficiently to prevent disorientation of the metal flakes on being removed from the field. C. O. C.

Flatting Agents. IC. *USP* 2,568,599

A tough, non-crazing, solvent-resistant flatting agent consists of the aluminium or other insoluble soap of an adduct of an $\alpha\beta$-unsaturated acid, e.g. maleic acid, and a terpene. Preferably the particles of this agent are coated with an originally water-soluble lacquer-insoluble vehicle, e.g. methyl cellulose or egg albumen. C. O. C.

Aqueous Polyvinyl Acetate Emulsion Paints. Shawinigan Products Corpn. *USP* 2,567,678

The process of *USP* 2,444,396 (J.S.D.C., **65**, 192 (1949)) is modified so as to produce a paint with better adhesion characteristics, hiding power, washability, paintability, and film-forming properties. C. O. C.

Synthetic Resin Coatings having a Polychromatic or Metallic Appearance. DuP. *BP* 667,982

Coating compositions which yield a uniform, unmottled surface without flocking or "flooding" when sprayed comprise an oil-modified alkyd resin vehicle, aluminium powder, and a β-dialkylaminoalkyl methacrylate. C. O. C.

Unsubstituted Ethylene Polymer Coating Compositions. DuP. *USP* 2,567,108

Incorporation of chromic acid into an aqueous dispersion of an unsubstituted ethylene polymer yields a composition which when applied to non-porous surfaces and baked has as good adhesion as that of conventional organic films. C. O. C.

Protective Coatings for Ferrous and Non-ferrous Metals. Industrial Metal Protectives. *BP* 669,736

A composition for protecting metals, especially iron, aluminium, or their alloys, from corrosion contains a resin of acid number not > 90, drying oil, solvent, drier, and zinc dust of average particle size not > 3 μ. and of which not < 90% can pass through a U.S. Standard 400-mesh sieve. C. O. C.

Moisture-setting Printing Inks. Sun Chemical Corpn. *BP* 669,987

A moisture-setting ink readily removed in wash-up operations by aqueous solutions consists of a water-immiscible resinous binder dissolved or dispersed in a water-miscible glycol, polyglycol, or glycol or polyglycol ester or ether and a partial ester or ether of a polyhydric alcohol with a long-chain fatty acid or alcohol or a condensate of ethylene oxide or polyethylene oxide with a long-chain fatty acid, amide, ester, alcohol, or thiol. C. O. C.

Polyvinyl Butyral Organosols—Coating Compositions (III p. 218).
Pigments for Printing Inks (IV p. 223).
White and Grey Silicon Nitride Pigments (IV this page).

VI—FIBRES; YARNS; FABRICS

Static Problems and their Control in the Textile Industry. J. A. Lopez and J. K. Hewson. *Amer. Dyestuff Rep.*, **41**, P 105–P 109 (18 Feb. 1952).

Reasons for the increasing seriousness of the static problem in textile processing, and its detrimental effect, are discussed. Methods of earthing, humidity control, chemical antistatics, radioactive salts, and high-voltage discharge bars are described. A detailed description is presented of an electronic static-control instrument, developed on the principles of controlled diffuse discharge. J. W. B.

Morphology of the Cotton Fibre. II—Bacterial Breakdown of the Cotton Fibre, and the Structural Elements of the Secondary Wall. W. Kling and H. Mahl. *Melliand Textilber.*, **33**, 32–37 (Jan. 1952).

An electron-microscopic study is made of cotton that has been given controlled treatments with a culture of flax-retting bacteria and with solutions of a pectinase and a cellulase. The enzymes, which are particularly effective when mixed, have a progressive etching action on the fibre. They reveal the fibrillar network in the primary wall and show that between the primary and secondary walls there is a homogeneous, resistant intermediate layer, similar to that covering the primary wall and possibly of similar constitution. The secondary wall contains groups of 10–20 fibril bundles, and each fibril bundle (diam. 0·1–0·2 μ.) consists of 100–200 single fibrils (diam. 100–200 Å.). The fibrils lie parallel to the bundle axis, and the bundles are inclined to the fibre axis and are arranged in the well known spiral fashion. The character of the individual bundles and fibrils is revealed very clearly by an examination of the breakdown material resulting from bacterial action. The groups of fibre bundles are separated by regions of open structure, which penetrate into the fibre radially. These, being particularly vulnerable to attack, develop into longitudinal grooves that can be observed with an ordinary microscope. Further attack in these regions leads finally to the disintegration of the fibre. A. E. S.

Chemical and Mechanical Preparation and the Spinning of Flax Waste. G. Thieme. *Melliand Textilber.*, **33**, 19–20 (Jan. 1952).

The chemical preparation of flax waste, which is usually done by boiling in an alkaline soln. in open or closed kiers for several hours, can be done more economically and with better removal of pectin material by immersing, after preliminary mechanical treatment to remove the greater part of the woody matter, in a cold alkaline soln. and then

boiling at low temp. under vacuum for 1 hr.; this is followed by souring, thorough washing, and drying. Some description is given of the mechanical preparatory treatments and of the carding and spinning operations necessary for the production of blends with rayon staple and worsted. A. E. S.

Structure of Jute. I X-Ray Diffraction Pattern. M. K. Sen and R. R. Mukherjee. *J. Textile Inst.*, **43**, P 114–P 121 (Feb. 1952).

A brief survey of the microscopic and chemical structure of jute and of X-ray studies on cellulose precedes an account of methods used in X-ray studies of jute and of the interpretations of the results obtained. Characteristic features are noted and compared with those of the ordinary cellulose diagram typified by that of ramie. It is not considered possible to use X-ray diagrams in classifying and grading different qualities of jute, a table being presented to show how similar diffraction characteristics are obtained from jutes of widely different qualities. J. W. B.

Electronographic Investigation of the Natural Cellulose, Ramie. A. L. Zaydes and I. G. Sinitskaya. *Doklady Akad. Nauk S.S.S.R.*, **80**, 213–214 (11 Sept. 1951).

Electron-diffraction measurements on cellulose have been carried out hitherto on regenerated films. However, ramie, in the form of fibrils obtained by dissecting the fibre under the microscope, gives a sharp electron-diffraction pattern. This and the X-ray pattern are shown. They are almost identical, but the X-ray pattern is more diffuse to an extent that is entirely in accord with a theoretically derived equation involving the relative wavelengths of the electron beam and the X-rays. A. E. S.

New Ways of Obtaining Fibre from Sisal. J. G. Theme. *Melliand Textilber.*, **33**, 1–4 (Jan. 1952).

The proportion of sisal fibre that passes into the waste may be very high, particularly in E. Africa, where the leaves, irrespective of their length, are passed through a standard decortication machine. It averages $\sim 25\%$. Mechanical extraction of fibre from waste leads to serious fibre damage; chemical separation is too dear; and biological retting does not occur by the method used for flax and jute. In the method of retting introduced by the author into Java in 1924, the waste is steeped in running air-saturated water. With a sufficiently high rate of flow (30 litres/sec. per metric ton), retting is complete in 3–4 days; at lower rates of flow, a longer time is necessary. Prolonged treatment leads to over-retting. The removal of soluble fermentable material, the maintenance of a non-acidic reaction, and Java climatic conditions are among the factors that make the process favourable for the selective survival of the retting bacteria, which belong to the *B. subtilis* and *mesentericus* group. In applying the principle of the method under the conditions of climate and water supply found in E. Africa, the author met with difficulties. They were overcome by treating the waste in the form of a layer, 1 m. deep. Water is sprayed on the bed during the day, 2·5 litres/sec. per metric ton being sufficient. In spite of the low temp., the exothermic fermentation reaction is sufficient to maintain the necessary temp. during the night. A 10-day treatment is given, and, working on an annular system, the bed is built up continuously at one end, as retted material is removed from the other. As retting proceeds, the mass compacts, and the resistance to the entry of air and water increases until the process comes to a standstill; over-retting is therefore impossible. Fibre produced by retting waste is softer than ordinary sisal fibre and is not irritating to the skin (removal of calcium oxalate). It has a staple of 24–30 cm. It is used for stuffing cushions, etc., and also for spinning cheap yarns for making sacks and coarse fabrics. A. E. S.

Sorption of Water Vapour by Cellulose. B. Grinberg. *Ann. Chim.*, **6**, 489–535 (1951): *Chem. Abs.*, **46**, 1251 (10 Feb. 1952).

The sorption isotherm of water vapour on cellulose is S-shaped and shows hysteresis. Its course cannot be predicted by any known equation and the mechanism of sorption is unknown, but sorption occurs in crystalline as well as in amorphous regions. Hydrolytic and ultrasonic degradation decreases the sorption, but extension increases it slightly. C. O. C.

Orientation in Cellulose Fibres as derived from Measurements of Dichroism of Dyed Fibres. P. H. Hermans and D. Heikens. *Rec. Trav. chim.*, **71**, 49–55 (Jan. 1952).

The dichroism of a number of viscose monofils (spun from ammonium sulphate), of varying degrees of stretch and dyed with Chlorazol Sky Blue FF, has been measured, and a dichroic orientation factor (f_D) obtained from the equation—

$$f_D = k\,\frac{E_{\parallel} - E_{\perp}}{E_{\parallel} + 2\,E_{\perp}}$$

(k = const.). The values of f_D thus obtained are always lower than the corresponding orientation factors obtained by X-ray (f_X) or birefringence (f_O) methods. Preston's suggestion (J.S.D.C., **62**, 368 (1946); **66**, 361 (1950)) that f_D represents a measure of the overall orientation of the entire fibre is not substantiated, as the ratio f_X/f_D is almost constant. A. J.

Correlation between Sensitivity to Acid Hydrolysis and Skin Core Differentiation in Viscose Rayon. P. H. Hermans and D. Heikens. *J. Polymer Science*, **8**, 187–190 (Feb. 1952).

The extent of hydrolysis under a given set of conditions is qualitatively related to the thickness of the fibre skin. Degree of orientation has little, if any, influence. C. O. C.

Membrane Potentials at Oxidised Cellophane. P. Hirsch. *Rec. Trav. chim.*, **71**, 354–360 (March 1952).

The difference Δ between membrane potential (M.P.) and liquid junction potential for KCl and NaCl soln. of different concn. separated by normal cellulose membranes and membranes oxidised with Br_2 and Na_2CO_3 has been measured at varying pH. Δ generally increased with pH to a max. at ca. pH 4, and is greater for oxidised than unoxidised cellulose. M.P. reaches half its max. value at pH 3–3·5. The shape of Δ-pH curves is analogous to a simple acid dissociation curve, and the expression—

$$\varepsilon = (\Sigma z_K - \Sigma z_A)\,\frac{RT}{F}\ln\frac{C_1}{C_2}$$

is derived, where ε = M.P. and z is defined as a "passage number", analogous to a Hittorf transport number through the membrane. For oxidised cellulose, carboxyl ions are fixed and $z_A = 0$. This treatment is too simple, and modifications are discussed. A. J.

Various Phase Equilibria in Cellulose Acetate Solutions. D. G. Dervichian and C. Magnant. *Rec. Trav. chim.*, **71**, 80–87 (Jan. 1952).

The system cellulose diacetate–chloroform–ethanol has been investigated at all concentrations of the constituents, and a general phase diagram obtained. Seven different regions of equilibrium are obtained, viz.—(1) First region of swelling, of two phases, ethanol concn. 0·8–1·6%, acetate concn. in swollen phase not > 12–13%. (2) First region of coacervation at ca. 2·5% ethanol. The concn. of acetate in the coacervate decreases as the ethanol concn. increases, but increases as the total acetate concn. increases. (3) Three-phase region between (1) and (2). (4) Single-phase region of soln. at 2·5–41% ethanol and up to 30% acetate. This phase is optically isotropic. (5) Single anisotropic phase showing birefringence, on the low alcohol concn. boundary of (4). (6) Second region of coacervation at ca. 40% ethanol and 0·5–13% acetate. The concn. of acetate in the coacervate increases as the ethanol concn. increases. (7) Second region of swelling. Increasing ethanol concn. beyond (6) gives swollen cellulose acetate with pptn. at 45–100% ethanol. Max. soln. of acetate occurs in region (4) at ca. 13% ethanol, corresponding to a max. osmotic pressure as observed by Dobry (*Bull. Soc. Chim. biol.*, **22**, 75 (1940)). A. J.

An Electron Microscope Study of the Surface Structure of Wool. R. L. Elliott and B. Manogue. J.S.D.C., **68**, 12–14 (Jan. 1952).

Epicuticle of Wool. G. Lagermalm. *Medd. Svenska Textilforskningsinst.*, (14), 65–69 (1951): *Chem. Abs.*, **46**, 2302 (10 March 1952).

The epicuticle of wool as distinguished from the middle exocuticle and the inner endocuticle is the outer membrane, 50–250 Å. thick, differing from the other two in both chemical and physical structure. With its relatively high resistance to hydrolysis by chemicals and enzymes and with its negative electrostatic charge, it offers both a

mechanical and an electrical barrier to negatively charged dyes. Treatment of wool with various agents, e.g. 1% aq. Na_2S, alcoholic KOH, bromine water, or phenoltrypsin, breaks down the inner membranes and leaves the epicuticle intact. Electron-photomicrographs show a surface with a network like that of orange peel. More severe digestion produces perforations in the film, enabling dyes to penetrate more easily. Sharp separation of the epicuticle from the other membranes is difficult to obtain by chemical reaction, but is effected naturally by the enzymes secreted by clothes moth grubs. Specimens present in their excreta viewed under the electron microscope, unlike those isolated by chemical means, are little damaged and have a striking and sharply defined surface pattern. The morphology of hen feathers is analogous to that of wool, and similar results were obtained with them. The nature of the X-ray interference pattern of the epicuticle suspension varies with the hydrolytic agent used. C. O. C.

Action of β-Propiolactone on Wool. C. Fearnley and J. B. Speakman. *J.S.D.C.*, **68**, 88–91 (March 1952).

Green Hair—a Contribution to the Problem of the Deposition of Metals in the Wool Fibre. A. Schöberl. *Melliand Textilber.*, **33**, 4–14 (Jan. 1952).

The effect of treating wool with hot water (90°C.) for several days in absence and in presence of various finely divided metals (Pb, Cd, Zn, Fe, Cu, Bi, and Hg) is followed by analysis of the wool for total S, cystine S, and the non-cystine S that is convertible into H_2S by HI reduction; the metal content of the wool and the nitrogen content of the soln. are determined also. Considerable amounts of metal may be taken up by the wool under these conditions, e.g. in 10 days—4·1% Pb, 7·3% Cd, 7·6% Zn, 8·0% Fe, and 10·2% Cu, and the wool becomes deeply coloured. The deposition of Pb, Cd, Zn, or Cu is accompanied by a considerable fall in the cystine content of the wool and a great rise in the nitrogen content of the bath, as compared with the control (metal absent). X-Ray analysis reveals the presence of metal sulphide (not always in its usual crystalline form) in all the treated samples, apart from the wool treated in presence of Cu; the latter gives no distinct pattern, but other considerations indicate that Cu_2S is indeed present in an amorphous form. In all cases, the amount of metal taken up is greatly in excess of the equiv. of the non-cystine S present, and some of it must be bound by functional groups of the keratin, including those formed by disulphide breakdown. Copper-treated wool is at first dark brown, but on exposure to air and light it slowly turns green. This is attributed to autoxidation with the formation of cupric compounds. A similar process can occur occasionally in the hair of copper-mine workers. Copper is deposited in the hair in presence of ore dust and sweat, and on further exposure the hair turns green. The spectroscopic estimation of metals in wool is discussed. The digestion of copper-treated wool by cyanide-activated papain is examined. Although copper compounds are poisons for papain, they do not interfere in presence of cyanide, owing to complex formation. A. E. S.

Swelling Studies of Single Human Hairs. M. G. Eckstrom. *J. Soc. Cosmetic Chem.*, **2**, 224–229 (1951): *Chem. Abs.*, **46**, 1079 (10 Feb. 1952).

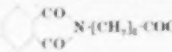
A special microscope slide cell, in which a single hair can be mounted, is fitted with inlet and outlet tubes, so that the hair can be treated with various reagents. From observations on the swelling of hair by NH_4 thioglycollate it is concluded that penetration of the thioglycollate solution is rapidly followed by reduction of disulphide bonds to form thiol groups and that the cystine concentration is almost constant throughout the cross-section of the hair. C. O. C.

Textile Fibres from Synthetic Textiles. G. M. Gantz. *Amer. Dyestuff Rep.*, **41**, P 100–P 104 (18 Feb. 1952).

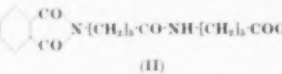
Physical, chemical, and dyeing properties of the newer synthetic fibres are discussed, with emphasis on dynel, Orlon, Acrilan, and Terylene. J. W. B.

Synthesis of N-6′-Aminohexanoyl-6-aminohexanoic ("Di-ε-aminocaproic") Acid. G. M. van der Want and A. J. Staverman. *Rec. Trav. chim.*, **71**, 379–384 (March 1952).

ε-Aminocaproic acid is condensed with phthalic anhydride to give phthalimidocaproic acid, which with thionyl chloride or PCl_5 gives the corresponding acid chloride (I). This condenses with ε-aminocaproic acid in dioxan-water as solvent in the presence of K_2CO_3 as condensing agent, yielding the phthaloyl-protected dipeptide (II). The phthaloyl group is removed as insol. phthalohydrazide by alcoholic hydrazine hydrate followed by acetic acid, and the "di-ε-aminocaproic" acid (III) is precipitated by addition of acetone. ε-Aminocaproyl-anilide is prepared in a similar way from I and aniline.



(I)



(II)

$H_2N \cdot [CH_2]_5 \cdot CO \cdot NH \cdot [CH_2]_5 \cdot COOH$

(III)

A. J.

PATENTS

High-tenacity Regenerated Cellulose Staple Fibres. Celanese Corpn. of America. *BP* 667,147

A tow of high-tenacity cellulose acetate filaments is breaker-drafted into staple fibre and saponified to convert the ester into regenerated cellulose. W. G. C.

Viscose Rayon. American Enka Corpn. *USP* 2,572,217

Milkiness in the filaments and incrustation of the orifices of the spinneret are avoided by using an acid coagulating bath containing a long-chain aliphatic tertiary amine–alkylene oxide condensate. C. O. C.

Fluorescent Coloured Artificial Fibres. BrC. *BP* 669,028

Fibres having the same colour when irradiated by light or by ultraviolet radiation contain a finely divided fluorescent inorganic pigment and a non-fluorescent organic dye of good fastness to ultraviolet radiation, whose colour in light matches that given by the inorganic pigment in ultraviolet radiation. C. O. C.

Casein Keratin Fibres. Courtaulds. *BP* 667,115

Protein fibres are obtained by spinning a mixture of casein and keratin dissolved in aqueous sodium sulphide into a salt coagulating bath of pH 6·0–9·0. W. G. C.

Hardening Casein Fibres with Titanium, Zirconium, or Tin Salts. Borden Co. *USP* 2,567,184

Fibres of high resistance to hot acids and alkalis and of light colour, good tensile strength, and even dyeing properties are produced by extruding alkaline aqueous casein into an acid coagulant, hardening in a second bath containing formaldehyde, and then without intermediate drying treating with an aq. soln. of a Ti, Zr, or Sn salt at pH 0·8–3·0 and 120–200°F. C. O. C.

Zein Fibres. Corn Products Refining Co. *BP* 667,283

Fibres of good dry strength are obtained by extruding an aqueous alkaline solution of zein into an aqueous saline coagulating bath and then, without precuring, stretching in a more concentrated aqueous alkaline saline bath. W. G. C.

Diamines and Polyamides. ICI. *BP* 667,771

Diamines of formula $H_2N \cdot CHAlk \cdot Ar \cdot CHAlk \cdot NH_2$ (Alk of 1–20 C; Ar = alkyl-subst. or unsubst. phenylene or diphenylene), e.g. *pp*′-bis-1-amino*iso*butyldiphenyl, from which may be produced soluble fibre-forming linear polyamides having low water absorption and satisfactory physical properties, are obtained either by catalytic hydrogenation in presence of ammonia of aromatic compounds containing two carbonyl groups, in each of which the C atom is directly attached to the nucleus and to Alk, or by converting the diketo compounds into the corresponding dioximes by treatment with hydroxylamine and then reducing with H_2. W. G. C.

Fluorinated Polyamides. American Viscose Corpn. *USP* 2,570,180

Fibre-forming polyamides having the repeating structural unit $-NH \cdot CH_2 \cdot [CX^1_2]_m \cdot CH_2 \cdot NH \cdot CO \cdot [CX^2_2]_n \cdot CO-$ (m = 0–4; n = 1–5; X^1 = F when m = 0; or X^1 and X^2 = H or F, at least one of them being F when $m > 0$) are obtained by condensing acid derivatives of formula $Y \cdot [CX^2_2]_n \cdot Y$ (Y = COCl or COOAlk) with diamines of

formula $H_2N{\cdot}CH_2{\cdot}[CX'_2]_m{\cdot}CH_2{\cdot}NH_2$. They have a regain of approx. 4%, are relatively inelastic, do not tend to creep or flow under constant load, have a waxy handle, are flameproof, and because of their density are suitable for making heavy fabrics. C. O. C.

Polyvinyl Alcohol Fibres. Comptoir des Textiles Artificiels. *BP* 667,582

Fibres of low salt content are obtained by extruding aq. polyvinyl alcohol into aq. ammonium sulphate, drying, and passing through a tensioning device which scrapes away from the surface of the fibres the bulk of the adhering salt. W. G. C.

Acrylonitrile Fibres. Industrial Rayon Corpn. *USP* 2,570,200–1 *USP* 2,570,237 *USP* 2,570,257

The continuous wet spinning of acrylonitrile fibres using ethylene carbonate as the solvent is described. C. O. C.

Tetramethylurea as Solvent for Polyvinylidene Cyanide. B. F. Goodrich Co. *USP* 2,574,369

Polyvinylidene cyanide is readily soluble in tetramethylurea to yield solutions which can be used for spinning or casting fibres or films. C. O. C.

Imparting Dimensional Stability to Polythiourea Fibres on Heating. Courtaulds. *BP* 669,059

Immersion of polythiourea fibres in an aq. solution of an aldehyde improves their dimensional stability on heating. C. O. C.

Coloured Glass Fibres. Owens-Corning Fiberglas Corpn. *USP* 2,569,700

Glass rod is coated with a vitreous enamel or other colouring matter which withstands the melting temperature of the glass, and the enamel fused into its surface, after which the glass is drawn into fibres. Alternatively the colouring matter is applied to fibres which are already partly drawn, and drawing is then completed. C. O. C.

Degradation and Dissolution of Viscose Staple Rayon during Sulphur Dyeing (VIII p. 228).
Potentiometric Investigations of Cuprammonium Solutions of Cellulose (XI p. 230).
Thin Membranes in the Surface Layers of Human Skin and Finger Nails (XII p. 231).
Ternary Copolymers (XIII p. 232).
Microkinematics of Textile Processes—Process of Stretching and Breaking Wool (in the Double Loop Test) (XIV p. 233).
Molecular Weight and Viscosity of Polyamides (Perlon, Nylon) (XIV p. 234).

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Rôle of Foam in Detergent Action. D. G. Stevenson. J.S.D.C., **68**, 57–59 (Feb. 1952).

Adsorption of Detergent by Cotton Sheeting under Textile Processing Conditions—Quantitative Data. L. H. Flett, L. F. Hoyt, and J. Walter. *Amer. Dyestuff Rep.*, **41**, P 139–P 143 (3 March 1952).

Adsorption of synthetic detergent (Nacconol NR) on cotton sheeting is studied by analysing the concentration of detergent in the liquor before and after treatment, using the partition end-point with chloroform. Effects of concn., time, temp., and pH are investigated. An interesting result is that the adsorption increases sharply with increasing concn. up to a certain point, then falls sharply to about half the maximum, and finally rises to a maximum again. This is considered to be due to variations in the colloidal state of the detergent. Up to 0·3% of pure organic sulphonate, based on fabric wt., may be adsorbed, necessitating care in maintaining the strengths of continuous-treatment baths. The desirability or otherwise of retaining detergent in the fabric is also discussed. J. W. B.

Removal of Textile Oils containing Mineral Oil and the Influence of the latter on Dyeing. H. R. Hirsbrunner. *Bull. mens. ITERG (Inst. tech. Études et Recherches Corps gras)*, **5**, 355–365, 414–434 (1951): *Chem. Abs.*, **46**, 1261 (10 Feb. 1952).

Discussion of the physical and chemical effects of textile oils on wool and of scouring treatments which remove mineral oil, the action of emulsifiers and detergents, and dyeing in presence of mineral oils is followed by an account of scouring trials to discover the efficacy of several Swiss commercial detergents in the removal of mineral oil. C. O. C.

Textile Application of Brighteners. O. L. Sherburne and J. P. G. Beiswanger. *Amer. Dyestuff Rep.*, **41**, P 144–P 148 (3 March 1952).

The use of fluorescent brightening agents on partly bleached muslin during soaping and during peroxide bleaching is discussed, and reference made to the possibility of their use with wool. An instrument is described for photometric measurement of brightening, whereby the ultraviolet reflectance is measured (*a*) with a filter passing only ultraviolet and not the fluorescence and (*b*) with one passing the fluorescence but not the ultraviolet radiation. These measurements are used to evaluate, under conditions of actual use, the strength ratios, selling values, and stability to other chemical treatments of brighteners. J. W. B.

Peroxide Bleaching of Cotton Yarns and Fabrics. T. E. Bell. *Amer. Dyestuff Rep.*, **41**, P 79–P 82 (4 Feb. 1952).

Basic methods and formulæ for bleaching cotton yarn, cloth, and knitwear are outlined, and an installation for continuous one-stage peroxide bleaching of cotton knit goods, of capacity 2500 lb./hr., is described in detail. J. W. B.

Characteristics of Cotton Fabrics processed in Continuous Peroxide Bleaching Systems. T. E. Bell and N. J. Stalter. *Amer. Dyestuff Rep.*, **41**, P 110–P 114 (18 Feb. 1952).

Sixteen samples of cotton cloths from different continuous peroxide bleaching installations were analysed in the grey, at intermediate stages, and after bleaching, for whiteness, fluidity, absorbency, non-cotton content, oil, fat and wax content, ash content, and pH. Whiteness and whiteness retention can be made commercially acceptable without fibre damage as determined by fluidity measurements. Absorbency and purity of cellulose are as good as in batch processing; pH values of the material are slightly higher, apparently without detriment. The function of the grey sour is held to warrant further study, as it has an effect on the oil, fat, and wax content, thus affecting indirectly the absorbency and whiteness. J. W. B.

PATENTS

Cleaning Raw Wool with Heavy Oil. Svenska Textilforsknings-Institutet. *BP* 668,467

Natural grease and other impurities are removed from raw wool by treating with a heavy oil having a flash point above that of the ambient temperature. Most of the oil is removed, preferably by centrifuge, and the wool carded without further lubrication. J. W. B.

Cleaning Undyed Materials contaminated with Heavy-metal Oxides or Water-insoluble Heavy-metal Salts. Ciba. *BP* 669,281

The material is treated with an alkaline solution of a salt of a nitrogen-free organic acid capable of forming water-soluble complex heavy-metal compounds, e.g. Rochelle salt, and a hydrosulphite. C. O. C.

Removing Graphite from Nylon. American Textile Co. *BP* 669,018

Aqueous dispersions of bentonite are highly effective in removing graphite from nylon, 1–10% dispersions giving the best results. C. O. C.

VIII—DYEING

Modifying Dyeing by Sound Waves—I and II. H. Rath and H. Merk. *Melliand Textilber.*, **33**, 211–216 (March); 311–314 (April 1952).

Fabrics composed of cellulosic fibres (**I**), or of wool, silk, acetate rayon, Perlon, or PeCe fibres (**II**) are dyed in an unstirred dyebath with various types of dye in presence and in absence of audible sound waves, produced by a Bosch generator placed in the dyebath; dyeing is done with and without agitation of the material. The presence of sound results in increased dyeing rate, penetration, and levelling. The effect on stationary material is generally comparable to the effect of agitation, the dyeing of agitated material being improved still further by simultaneous

sound treatment. It is considered that the low efficiency and short life of existing sound generators make the method impracticable on the large scale. The use of ultrasonic devices is still more impracticable. A. E. S.

Theory of Dyeing Processes. E. Elöd. *Textil-Praxis*, **7**, 66–69 (Jan. 1952).

Published work dealing with fundamental reactions taking place in the dyeing of wool, silk, nylon, and Ardil is reviewed together with theoretical considerations which can be deduced from these reactions. B. K.

Heats and Free Energies of Formation of some Hydrogen Bonds. M. St. C. Flett. J.S.D.C., **68**, 59–64 (Feb. 1952).

Package Dyeing. I—A Theoretical Model and its Relation to Technical Practice. J. Boulton and J. Crank. J.S.D.C., **68**, 109–116 (April 1952).

Degradation and Dissolution of Viscose Staple Rayon during Sulphur Dyeing. G. Nitschke. *Textilpraxis und Textiltech.*, **3**, 66–72 (Feb. 1952).

The solubilities of two types of viscose staple fibre (in the oiled state) in typical "blank" sulphide dyebaths are determined and tabulated. The concn. of Na_2S (5–20 g. per litre), the temp. (20–95° c.), and the time (0·5–60 min.) are varied. Also, Na_2S is replaced by K_2S. Under the usual dyeing conditions, 2–3% of the rayon is dissolved. Staple viscose sliver is dyed with Sulphur Black AW and with Sulphur Yellow R (Wolfen). Nine alternative treatments after dyeing include—rinsing very thoroughly in soft water and drying; drying without rinsing; and drying, with or without rinsing, followed by impregnation in dil. sodium carbonate soln. and drying. The alkali solubility and fibre tensile properties are determined on the finished material. Results for the black dyeing show that drying without rinsing leads to appreciable degradation (attributed to the formation of sulphuric acid in the fibre), but that this is greatly reduced when the sodium carbonate treatment has been given. Material that has been thoroughly rinsed after dyeing suffers least—its fibre strength is actually higher, and its alkali solubility lower, than for the untreated material, but a "loop" tensile test shows that some embrittlement has occurred during dyeing. Only small degradation effects are observed for the yellow dyeing. It is recommended that dyeing should be followed by very thorough rinsing, and that, if the precaution of finishing in an alkaline soln. is taken, then sodium acetate or formate should be used, rather than carbonate. A. E. S.

pH Control of Acid and Chrome Dyeing. II—Single-bath Mordant Dyeing. G. H. Lister. J.S.D.C., **68**, 49–57 (Feb. 1952).

Mordants used for Oxidation Dyes. I—Action of Mordants in Dyeing. II—Relation between the Mordant and the Weakening of the Dyed Material. S. Yamaguchi and H. Iida. *Repts. Govt. Chem. Ind. Research Inst. Tokyo*, **45**, 261–269 (1950): *Chem. Abs.*, **46**, 1769 (25 Feb. 1952).

I—Rabbit fur impregnated with a metallic mordant was dyed with *p*-phenylenediamine in 0·3% H_2O_2. The amount of diamine absorbed and the rate of its decomposition by H_2O_2 indicated that the mordant did not fix but catalysed the oxidation of the diamine. $CuSO_4$, $FeSO_4$, and $(NH_4)_6Mo_7O_{24}$ were effective mordants; $K_2Cr_2O_7$ was not too effective; alum, $NiCl_2$, $Cr_2(SO_4)_3$, and $MnCl_2$ were only slightly effective. When the fur was dyed with *p*-aminophenol the metallic salts acted as fixers and catalysts.

II—The tensile strength of rabbit fur was unaffected by impregnation with $K_2Cr_2O_7$, K_2CrO_4, or $KMnO_4$, but decreased to about 60% when the impregnated fur was dyed with *p*-phenylenediamine. This weakening could be avoided by painting the flesh side of the pelt with $Na_2S_2O_3$ and acetic acid after the mordanting, and rinsing before dyeing. C. O. C.

Dyeing Nylon, Orlon, and Terylene. S. G. Turnbull. *Amer. Dyestuff Rep.*, **41**, P 75–P 78, P 82 (4 Feb. 1952).

A survey of the dyeing methods for these three types of fibre. J. W. B.

PATENTS

Phloroglucinol as Developer for Diazotisable Dyes. C. E. Maher. *USP* 2,546,861

Phloroglucinol may be employed instead of the widely used 2-hydroxy-3-naphthoic acid as the developer for diazotisable dyes on acetate rayon, nylon, cotton, or viscose provided that excess nitrous acid from the diazotisation process is removed by having substances like urea, sulphamic acid, or melamine in the coupling bath. A suitable bath for developing a 2·5% dyeing of the acetate rayon diazo black base *p*-aminophenylazobishydroxyethylaniline, after diazotisation on the fibre, contains 2 lb. sodium acetate, 2 lb. sulphamic acid, and 8 oz. phloroglucinol in 100–400 gal. (U.S.A.) water per 100 lb. fabric, at pH 5–7. Diazo direct cotton dyes, *e.g. C.I.* 401, are developed in a similar bath at pH 7. E. S.

Metachrome Dyeing. FBy. *BP* 668,598

The affinity of most metachrome dyes for wool is increased if non-ionic surface-active compounds, e.g. polyglycol ethers, and salts of magnesium or alkaline-earth metals are added to the dyebath. C. O. C.

Dyeing Furs and Skins. Ciba. *BP* 669,192

An aqueous bath containing not < 10% of a water-miscible low-molecular aliphatic alcohol and/or ketone and/or ester is used with dyes which are soluble in either water or the organic liquid. The method is especially suitable for dyes containing no sulpho or carboxyl groups, and in many cases is particularly advantageous for applying the metal complex compounds of azo dyes. Temperatures of 40–60° c. may be applied, and in most cases the hair is dyed much deeper than the skin, good penetration being obtained. C. O. C.

Colouring only One Component on the Face of a Fabric containing Two or More Fibres. Royal Swan. *USP* 2,573,705

In a fabric composed of mixed fibres one component only, and only that part of it which is present in the face of the fabric, is coloured by coating the face of the fabric with a dye liquor of such viscosity that it cannot penetrate the fabric and containing a dye having affinity for the component to be coloured, drying rapidly, and then vigorously washing. C. O. C.

Dyeing Moulded Organic Derivatives of Cellulose. Celanese Corpn. of America. *BP* 669,720

Moulded cellulose acetate or other organic derivative of cellulose is quickly dyed with deep penetration in an aqueous dispersion of a dye for the cellulose derivative, soap, and a water-soluble monoalkyl ether of mono- or di-ethylene glycol. C. O. C.

Green Hair—a Contribution to the Problem of the Deposition of Metals in the Wool Fibre (VI p. 226).
Removal of Textile Oils containing Mineral Oil and the Influence of the latter on Dyeing (VII p. 227).
Printing or Dyeing with Sulphur Dyes (IX p. 229).

IX—PRINTING

Fundamental Processes of Textile Printing. IV—Transfer of Acid Dyes to Cellulose during Steaming. R. B. Patel and H. A. Turner. J.S.D.C., **68**, 77–87 (March 1952).

Diffusion Phenomena in Multilayer Colour Photography. K. Meyer and H. Ulbricht. *Z. wiss. Phot.*, **46**, 72–83 (1951): *Science et Ind. phot.*, **22**, 469–471 (1951): *Chem. Abs.*, **46**, 1375 (25 Feb. 1952).

A study of diffusion effects of importance in colour photography. C. O. C.

Photodichroism of Dye Gelatin Films. I—Relaxation Phenomena. II—Correct Dichroism Time Curves considering Relaxation Phenomena. D. Yamamoto and R. Iwaki. *J. Chem. Soc. Japan, Pure Chem. Sectn.*, **72**, 852–854 (1951): *Chem. Abs.*, **46**, 1846 (10 March 1952).

I—Two relaxation times are necessary to account for the relaxation phenomena of photodichroism of gelatin films containing various dyes. The short one is due probably to the temporary dichroism in phosphorescent dye–rigid solvent systems as suggested by Lewis *et al.* (*Chem. Abs.*, **36**, 343 (1941)), the longer one to the anisotropic fading in the Weigert effect.

II—The photodichroism of dye-gelatin films shows rapid decay immediately illumination by incident light ceases. Instead of taking the value after decay, as has hitherto been done, the relaxation was taken into account to get correct photodichroism-time curves. The value of the dichroism, which usually increases with time of irradiation, passes through a maximum, and then decreases gradually, depends in a more complex manner on time after correction is made for the decay mentioned above. C. O. C.

Magnetic Dichroism of Dye Gelatin Films I. D. Yamamoto. *J. Chem. Soc. Japan, Pure Chem. Sectn.*, **72**, 693–695 (1951): *Chem. Abs.*, **46**, 1377 (25 Feb. 1952).

Dye-gelatin films show dichroism in a magnetic field of 5000–8000 gauss. C. O. C.

Chemical Toning of Photographic Prints. D. R. Dhingra, M. G. Gupta, and B. P. Singh. *J. Proc. Inst. Chemists* (India), **23**, 9–35 (1951): *Chem. Abs.*, **46**, 1378 (25 Feb. 1952).

A review of the chemistry of toning, including dye toning. C. O. C.

PATENTS

Printing or Dyeing with Sulphur Dyes. Vereinigte Färbereien. *BP* 669,002

Wool can be dyed or printed with sulphur dyes if the dye has been reduced with a "hydrosulphided" (by means of H_2S) ethanolamine in presence of an organic base to give the bath the necessary alkalinity. C. O. C.

Increasing the Wet Fastness of Material printed with Water-soluble Dyes. Courtaulds. *BP* 668,421

In printing with water-soluble dyes, bleeding of the dye during printing is avoided and increased fastness to washing of the prints is obtained by adding to the printing paste a water-insoluble cyanamide–formaldehyde condensate and an organic acid to bring the condensate into solution. C. O. C.

Printing Cloth or other Web-like Material. J. Dungler. *BP* 668,529

Deformation of the cloth during multicolour printing is avoided by causing it to adhere to a flexible inextensible support, which is attached to rollers running on laterally adjustable guides which permit the transverse tension in the support to be varied as necessary. C. O. C.

Transfer Sheets which can be handled without Smudging. Columbia Ribbon & Carbon Manufacturing Co. *BP* 669,212

A protective layer for the transfer coating comprises a binding agent containing an anti-friction shielding component of lamelliform particles. These particles, *e.g.* Al, mica, graphite, or fish scales, should preferably be coloured differently from the transfer layer. C. O. C.

Colour Photography. Ilford. *BP* 668,215

Colour formers containing sulpho radicals and a highly hydrophobic anion yield complexes with gelatin. Such complexes, containing also silver halide, may be dispersed in aqueous or organic solvents to produce photographic emulsions in which the colour former is ionically linked to the gelatin. C. O. C.

Producing Masked Colour Images in a Multilayer Colour Photographic Film. Kodak. *BP* 668,642

Four-colour Photography. E. M. Boudot and J. M. J. P. d'Andre. *BP* 668,647

Eliminating Stain in Colour Photography. Eastman Kodak Co. *USP* 2,571,725

Photosensitive Glass. Corning Glass Works. *BP* 668,767

A glass is described which on being exposed to short-wave, e.g. ultraviolet, radiation and then heat develops a colour in the exposed area but not in the unexposed area. C. O. C.

X—SIZING AND FINISHING

Shrink-resisting of Men's Half-hose. M. Lipson and C. H. Nicholls. J.S.D.C., **68**, 21 (Jan. 1952).

PATENTS

Intermittently applying Lubricant to Sized Yarn. I. L. Griffin. *USP* 2,568,446

As the warp passes from the size-box to the drying cylinder it is intermittently treated, say at intervals of 1 in., with a lubricant or other liquid. This liquid is in turn deposited on the drying cylinder to produce a film, which then covers the warp. This enables all softeners to be left out of the size. C. O. C.

Fugitive Tinting. Celanese Corpn. of America. *BP* 668,957

The fugitive dye is applied dissolved in a mixture of isobutyl alcohol (1–5%, by weight), water (90–97), and a sodium dialkyl sulphosuccinate (0·2–1·0). Such a solution does not form slime or jelly in the furnishing device. C. O. C.

Improving the Mechanical Properties of Regenerated Cellulose Fibres. M. R. Wirth. *BP* 669,088

Improved crease- and shrink-resistance, handle, and tensile and wet strength are obtained by treatment with a precondensate of dicyandiamide and an aldehyde at pH 6–7 followed by curing. If desired, urea and/or melamine may replace some of the dicyandiamide. C. O. C.

Permanent Glazed Finish. Monsanto. *BP* 669,185

A permanent glazed finish is obtained, without any loss of tensile strength or tendency toward yellowing, by treating with an aqueous solution or dispersion of an ammonium or amine salt of a copolymer of styrene or a substituted styrene with maleic anhydride or a partial ester of maleic acid, and then hot-calendering. C. O. C.

Permanent Stiff Finish. Monsanto. *BP* 669,187

A stiff finish resistant to washing is obtained by treatment first with an aqueous solution or dispersion of an alkali or amine salt of a copolymer of styrene or a substituted styrene with maleic anhydride or a partial ester of maleic acid and then with a dilute solution or dispersion of a heavy-metal salt. C. O. C.

Imitation Leather. A. Edlinger. *BP* 666,792

A textile fabric, e.g. cotton cloth, is thoroughly napped, and coated with natural or synthetic rubberlike materials so that only the tips of the nap fibres are covered by the first coat and are not flattened by it. Subsequent coats are applied, but the upright nap structure is preserved and results in a grain-like finished appearance, and also imparts a cushion effect which leads to excellent flexibility. J. W. B.

Bonding Yarns to Rubber. Courtaulds. *BP* 667,778

Better bonding between regenerated cellulose yarns and rubber is obtained if the yarns are first treated with glyoxal. C. O. C.

Flameproofing Cellulosic Textiles with Titanium Antimony Compounds. DuP. *USP* 2,570,566

A finish which passes the A.A.T.C.C. flash- and flame-resistance test is given by impregnation with an aqueous solution of Ti and Sb in an inorganic monobasic acid, there being not < 25 g. of the oxides of Ti and Sb per litre, the atomic ratio of Sb : Ti being not > 2, and the weight ratio of Ti : Sb being not > 10. C. O. C.

Rendering Wool Goods resistant to Felting and Shrinking. Textilwerk. *BP* 669,442

The materials are impregnated with an aqueous solution of melamine–aldehyde resin and formaldehyde at pH < 7·0, and are then dried, and stored for 4–6 weeks. During storage the resin hardens without need for baking or any special curing process. The treated goods have the same handle as untreated goods. C. O. C.

Imparting Wiriness to Wool. Fred Whitaker Co. *USP* 2,570,750

Soft South American wools are rendered more brash or wiry and so suitable for carpets or upholstery fabrics by degreasing the wool to < 0·75% grease content and then treating at any stage of manufacture with an aqueous dispersion of colloidal silica of initial particle size 10–100 mμ. and pH 2–6 until 0·1–2·0% silica on the dry weight of the wool has been absorbed. The brashness so imparted resists normal processing, including curling and dyeing. C. O. C.

Crush-resistant and Water-repellent Finish. Socony-Vacuum Oil Co. *USP* 2,569,695

Textiles, paper, fur, etc. are impregnated with an aqueous solution or dispersion of a nitrogenous phenolic resin (e.g. a phenol–aldehyde–ammonia condensate) and then with a water-soluble derivative of a nitrogenous heterocyclic resin (e.g. the condensate of formaldehyde, ammonia, and a 5-membered heterocyclic compound), and are finally heat-cured. C. O. C.

Papermakers' Felt. Orr Felt & Blanket Co. *USP* 2,567,097

High wettability, low wetting time, and high resistance to abrasion are imparted by first impregnating the felt with an aqueous solution of a synthetic resin, then, after curing and ageing the resin, impregnating the felt with an aqueous solution of synthetic rubber, and finally curing the rubber. C. O. C.

Stretchable Woven Fabrics. United States Rubber Co. *USP* 2,574,200

A fabric having heat-shrinkable yarns in the weft is heated so that it shrinks weftwise. The shrunken fabric is coated with tacky rubber, which is used to bind the fabric to a moving and longitudinally stretched belt. This belt is then allowed to contract, and the fabric dried while on the belt, so as to cause the rubber to hold it in its warp-shrunk condition. It is then stripped from the belt, and cured to yield a two-way-stretch fabric. C. O. C.

Felting Covers on Tennis Balls to produce a Homogeneous Seamless Felt Cover. R. G. Tugen. *BP* 668,746

Condensation of Formaldehyde with Urea (XIII p. 232).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Carbon Paper Manufacture. A. H. Woodhead. *Paint*, **21**, 123–129 (April 1952).

A general account of the materials and methods used in making the various types of carbon paper now available. C. O. C.

Potentiometric Investigations of Cuprammonium Solutions of Cellulose. M. J. Arkhipov and V. P. Kharitonova. *Faserforsch. und Textiltech.*, **3**, 35–37 (Jan. 1952).

A long abstract in German of a Russian paper, with tables and diagrams. See J.S.D.C., **67**, 471 (Nov. 1951). A. E. S.

Protection of Cellulose Esters against Breakdown by Heat and Light. G. C. DeCroes and J. W. Tamblyn. *Modern Plastics*, **29**, (4), 127–136, 185–189 (April 1952).

The plasticiser is often the chief contributor to breakdown by heat. Most common plasticisers are more easily oxidised than the cellulose ester itself, and the oxidation process induces breakdown of the chains. Antioxidants inhibit this type of breakdown, whereas various metal soaps and TiO_2 catalyse it. Where an oxidation-resistant plasticiser is present, a similar breakdown can be produced by addition of a peroxide. A severe but non-oxidative type of breakdown occurs when the plasticiser contains ferric salts of strong acids. This thermal breakdown can be greatly reduced by addition of a suitable chelating agent. In breakdown by ultraviolet radiation oxygen also plays an important part. The oxidisable plasticiser, dibutyl sebacate, contributes to this type of breakdown, but to a far lesser extent than to breakdown by heat. Ultraviolet-breakdown inhibitors are discussed, and it is shown that antioxidants are useful only if combined with an additive which efficiently filters out ultraviolet radiation. In studies of weathering inhibitors, the thickness of the test piece is an important variable. Mechanisms by which ultraviolet-breakdown inhibitors may be effective are briefly speculated on. 35 references. C. O. C.

PATENTS

Disinfecting Cellulosic Pulp. Procédés et Produits Chimiques Prochim S.A. *BP* 667,622

A suitable disinfectant is an organo-mercurial compound of formula $CH_3{\cdot}O{\cdot}C_2H_4{\cdot}Hg{\cdot}X$ (X = acid radical or halogen), e.g. methoxyethylmercury chloride. W. G. C.

Sizing Cellulosic Fibres with Cationic Melamine Resin and Hydrophobic Material. American Cyanamid Co. *USP* 2,563,897

Addition of cationic melamine–aldehyde resin to aqueous dispersions of water-insoluble coating or impregnating agents added to paper stock or the like causes controlled flocculation, so that the particles of the coating or impregnating agent are uniformly deposited on or in the fibres. C. O. C.

Coated Paper. D. R. Erickson. *BP* 667,437

Coated paper having a printing surface consisting of a continuous film of filler interspersed in binder is made by coating paper with filler (100 parts by weight) dispersed in a solution of binder (hydroxyalkyl cellulose) (6–10). The binder, which is insoluble in water, is dissolved in aq. NaOH, which is eliminated from the solution immediately after its application, thereby precipitating the cellulosic compound to bind the filler to the base, by treatment with aq. $(NH_4)_2SO_4$ or other water-soluble compound. The paper is afterwards dried in contact with a smooth surface. S. V. S.

Coating Paper. Mead Corpn. *USP* 2,565,260

Production of a mineral suspension, and a machine for applying it to paper so as to produce a mineral-finished surface of high smoothness and finish, are described. C. O. C.

Coating Paper. Chemical Manufacturing Co. *USP* 2,566,529

The amount of casein required for producing highly finished paper can be reduced by using a dispersion of a vegetable resin, e.g. resin or colophony, in aqueous alkaline casein solution. C. O. C.

Insecticidal Paper. Hercules Powder Co. *USP* 2,566,092

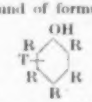
Paper is rendered insecticidal by impregnating it with a polychlorinated bicyclic terpene containing 40–75% combined Cl, e.g. chlorinated camphene of 68% Cl content. C. O. C.

Hydrophilic Film Sheets or Pellicles. British Sidac. *BP* 667,415

A transparent non-fogging sheet material suitable for containers or wrapping comprises a clear transparent hydrophilic pellicle having an inherently moistureproof coating consisting of a thermoplastic resin with 0·5–5·0% on its weight of a wetting agent to spread out droplets of condensed water into a continuous film. The resin may be a copolymer of vinylidene chloride and vinyl chloride of high viscosity, a polymer of vinylidene chloride, or cyclised rubber, and the wetting agent a fatty alcohol sulphate, an aralkylsulphonate, or a long-chain dialkyl ester of sulphosuccinic acid. The coating may be applied direct to the pellicle, or one or more intermediate water-resisting coatings may be applied. S. V. S.

Stabilising Cellulose Ethers to Heat. Hercules Powder Co. *USP* 2,564,581

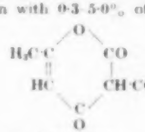
Ethyl cellulose and other thermoplastic cellulose ethers may be heated to 200°C. without discoloration in presence of SO_2 and a compound of formula—



(T = a cyclic sat. terpene, one H atom of which is substituted by the phenol; one R of the phenolic group is the terpene, the other Rs being H, or at least 2 H-atoms with the rest sat. hydrocarbon or alkoxyl groups). C. O. C.

Fungistatic Paper, Regenerated Cellulose Sheeting, etc. Dow Chemical Co. *USP* 2,567,010

Impregnation with 0·3–5·0% of dehydroacetic acid—



renders paper, etc. fungistatic. C. O. C.

Cast Coated Paper. Champion Paper & Fibre Co. *USP* 2,568,288

A continuous film of oil is formed on the clear casting surface, and is then reduced to monomolecular dimensions before casting begins. This results in high-quality coating and in the coated paper being readily, completely, and cleanly detachable from the casting surface. The process can be made continuous. C. O. C.

Pigment Binder Compositions for producing Mineral- or Pigment-coated Paper. Monsanto. *BP* 668,525

Paper of high brightness, high gloss, and good affinity for printing inks is obtained by coating paper with a mineral pigment slurried in an aqueous solution of a salt

of a styrene–maleic anhydride copolymer, or of an unsubst. or N-Alk- or N-Ar-subst. half amide of such a copolymer, and then calendering the dried coated paper. The pigment forms the greater part of the solids in the slurry, the amount of the salt used being 5–30% of the weight of the pigment. C. O. C.

Gelatin Baryta Coating for Photographic Papers. Kodak. *BP* 668,970

A dispersion of baryta in aqueous gelatin containing glyoxal as a hardening agent is an excellent coating composition for producing uniform coatings on photographic paper. C. O. C.

Coating of Paper. Consolidated Water Power & Paper Co. *BP* 670,414 *USP* 2,578,345

Paper and paper board are coated with an aqueous solution of a plasticisable, film-forming substance of resinous character, chemically inert to the fibres, e.g. polyvinyl alcohol, to which a converted starch may be added, and passed through rolls to plasticise and smooth the resulting coating on the surface; then, while still damp, they are imprinted with a thin film of a mineral-pigment coating of high consistency (not > 50% by weight of water) in an aqueous vehicle on the same side as the first coating, dried, and calendered.

BP 670,478

The first coating solution contains at least 5% by weight of converted starch. S. V. S.

Mineral- and/or Pigment-binding Composition for Coating Paper. Monsanto. *USP* 2,576,914

A pigment or mineral, e.g. blanc fixe or talc, is dispersed with the aid of a long-chain unsaturated fatty acid or a derivative thereof in an aqueous solution of a water-soluble salt of the copolymer of an alkyl half ester of an ethylene-$\alpha\beta$-dicarboxylic acid and/or anhydride and a compound of formula $R{\cdot}CH{:}CH_2$ (R = subst. or unsubst. phenyl). Such compositions give a coating of excellent adhesion, uniformity, and resistance to calendering.

USP 2,577,624

The binder used consists of a styrene–maleic anhydride copolymer. C. O. C.

Waterproofing Emulsions for Paper. R. C. Hoyes. *BP* 670,701

An aqueous emulsion of paraffin wax containing a water-soluble resinous soap and/or an alkali-metal salt of carboxymethyl cellulose and a chlorinated resin is used. The emulsion may contain also a water-soluble organic salt of Al; if it does not, then the emulsion-treated paper must be further treated with an aqueous solution of such a salt. C. O. C.

Sizing and Drying Paper as it passes to Roller Printing Machines. Hartleys (Stoke-on-Trent). *BP* 670,724

Moisture-proof Sheet Material. British Cellophane. *BP* 670,108

Moisture-proof sheet material is produced by depositing on a base film of water-sensitive, non-fibrous organic material, e.g. regenerated cellulose, a partly condensed urea–formaldehyde resin deposited from an aqueous dispersion containing no condensing agent, drying the resin-containing film without curing the resin, then applying a liquid moisture-proofing composition comprising a film-forming agent and a volatile organic solvent containing in solution an acidic condensing agent for the resin, and heating to remove the solvent and cure the resin. S. V. S.

Esters of Hydroxysulphones—Plasticisers (XIII p. 232).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Electron-microscopic Investigation of the Effect of Electrolytes on the Structure of Collagen. A. L. Zaydes and S. L. Pupko. *Doklady Akad. Nauk S.S.S.R.*, **80**, 647–648 (1 Oct. 1951).

Collagen is treated with solutions of acetic acid, hydrochloric acid, ammonium sulphate, and ammonium thiocyanate, and the changes in structure are observed by means of the electron microscope. Both acetic and hydrochloric acids at 0·01 N. lead to the breakdown of the fibrils into subfibrils, which are found to be in the form of an intricate network. At normal strength, acetic acid causes an apparent loss in structure, while HCl leaves the original fibrillar structure apparently unchanged. These results are in accord with the increased swelling in acetic acid with increase in concn., and with the reverse effect for HCl. Ammonium sulphate produces a loosening and opening up of the fibrillar structure, while ammonium thiocyanate causes a compacting of the structure with loss of the original striped appearance. A. E. S.

Thin Membranes in the Surface Layers of Human Skin and Finger Nails. G. Lagermalm, B. Philip, and J. Lindberg. *Nature*, **168**, 1080–1081 (22 Dec. 1951).

Thin pieces of skin loosened from sun-burnt human back, thickened skin from the palm of the hand, and finger-nail parings are treated for 6 days at 50°c. and 14 days at room temp. with 1% Na_2S. Electron micrographs of the washed and palladium-shaded residues show membranes wrinkled to a much higher degree than those from hairs and feathers, with wrinkles running in all directions. This may be due to the membranes having originally enclosed ellipsoidal cell structures rather than cylindrical structures.

Wool epicuticle shows signs of damage after treatment with hyaluronidase, but if wool itself is so treated no increase in acid sorption is found. J. W. B.

Lotmar–Picken X-Ray Diagram of Dried Muscle. C. H. Bamford and W. E. Hanby. *Nature*, **168**, 1085–1086 (22 Dec. 1951); **169**, 120 (19 Jan. 1952).

Observations are made on results obtained by Lotmar and Picken (*Helv. Chim. Acta*, **25**, 538 (1942)). J. W. B.

Lotmar–Picken X-Ray Diagram of Dried Muscle. L. Pauling and R. B. Corey. *Nature*, **169**, 494–495 (22 March 1952).

Comparison of Lotmar–Picken muscle X-ray diffraction photographs with those taken simultaneously from horse-hair and small needle-like urea crystals (the latter to simulate the crystalline zones) indicates that the muscle contains about 10% of crystalline material, which need not therefore be protein, as muscle contains about 10% of non-protein material. However, residue spacings are discussed in detail and the resemblance between the Lotmar–Picken diagram and that of poly-γ-methyl-L-glutamate is held to indicate that the crystalline portion is in fact a polypeptide of high mol. wt. J. W. B.

PATENTS

Leather. Forestal Land, Timber & Railways Co. *BP* 666,619

Leather is produced by treating hides first with non-resinous aldehyde–ketone condensates and then with alkali so as to convert the condensates into resins *in situ*. C. O. C.

Syntans from Vanillin. United States Leather Co. *USP* 2,564,022

Leather tanned at pH 3·5 with the product obtained by treating 2 mol. of phenol and 1 mol. of vanillin with conc. H_2SO_4 has a full and excellent feel. C. O. C.

Tanning. Société de Produits Chimiques des Terres Rares. *BP* 670,685

Excellent tanning is obtained by use of a complex soluble Zr salt dispersed in a silica gel using 0·1–2·0 mol. of silica gel per mol. of ZrO_2. C. O. C.

Tanning with Formals. United Shoe Machinery Corpn. *USP* 2,577,033

The dried skin or hide is treated with an organic-solvent solution containing a formal (2% by vol.) and a conc. aq. strong mineral acid (0·3–0·4% by vol.). The products withstand prolonged soaking in water without marked shrivelling or hardening even at high temp., and are exceptionally resistant to mildew. C. O. C.

Leather having a Suède Finish on its Flesh Side and a Printed Grain Side. Bolton Leathers. *BP* 670,927

Suède-finished leather which is printed on the grain side requires no lining when it is made into handbags, shoes, etc. After printing it is steamed for 1 hr. at 80°c., washed, treated in a 10% solution of a urea–formaldehyde resin, washed until all loose colour has been removed, and dried and finished as usual. C. O. C.

Photodichroism of Dye–Gelatin Films. I—Relaxation Phenomena. II—Correct Dichroism–Time Curves considering Relaxation Phenomena. (IX p. 228).

Magnetic Dichroism of Dye–Gelatin Films—I (IX p. 229).

XIII—RUBBER; RESINS; PLASTICS

Report on Nomenclature in the Field of Macromolecules. International Union of Pure and Applied Chemistry. *J. Polymer Science*, **8**, 257–277 (March 1952).

This report is divided into seven sections, of which II—General Definitions, III—Special Definitions, and VII—Linear High Polymers are of greatest importance to members of the Society. C. O. C.

Copolymerisation Theory of the Vulcanisation of Rubber. VI—Hydrogen Sulphide Effects and some Self-limiting Features. D. Craig, A. E. Juve, W. L. Davidson, W. L. Semon, and D. C. Hay. *J. Polymer Science*, **8**, 321–335 (March 1952).

Vulcanisation with sulphur is due to formation of sulphur radicals, which copolymerise with the double bonds of the rubber molecule. Hydrogen sulphide is necessary for chain initiation, but when sufficient is formed it acts as a chain terminator. This probably leads to numerous self-limiting features, including ceiling temperatures, post-vulcanisation, and delayed-action effects. Initiation may result from oxidation of H_2S to sulphur radicals and termination from formation of HS radicals. C. O. C.

Condensation of Formaldehyde with Urea. G. Smets and A. Borzee. *J. Polymer Science*, **8**, 371–394 (April 1952).

When formaldehyde is condensed with urea (molar ratio 1 : 1 and 1 : 2 of both components), the reaction always proceeds by a second-order mechanism. These reactions are analogous to the condensation of pure mono- and di-methylolurea. In neutral solution at 65, 75, and 85°C. the energy of activation is 21 kg.cal./mole for both reactions; nevertheless, the 1 : 2 ratio condensation is three times slower than the 1 : 1 condensation. In acid solution, a pure acid-catalytic effect occurs and the rates of reaction at pH 3–5 are proportional to $[H^+]$; in this case the activation energy is approx. 8–10 kg.cal. At higher pH, both thermal and acid catalytic actions occur simultaneously. The apparent kinetics of the reactions are given. C. O. C.

PATENTS

Variegated Colourings on Rubber. Sterling Rubber Co. *BP* 667,781

Variegated colouring is produced on thin rubber articles by compressing them in a crumpled state, and while compressed treating the exposed surfaces with a dispersion of a pigment in a dilute solution of rubber. C. O. C.

Plasticising Vinyl Resins. Monsanto. *BP* 661,380

Plasticisers for polyvinyl chloride and its copolymers, having good permanence and conferring good low-temperature flexibility, comprise alkyl (6–12 C) or alkoxyethyl (6–14 C) phenyl tolyl phosphates. E. C.

Esters of Hydroxysulphones—Plasticisers. Shell Refining & Marketing Co. *BP* 662,656

Thioether alcohols of formula $R{\cdot}S{\cdot}[CH_2]_n{\cdot}OH$ (R = Alk, *cyclo*alkyl, or Ar; $n > 1$) are esterified with mono- or di-carboxylic acids either before or after oxidation with hydrogen peroxide to the corresponding sulphones. The products, e.g. 2-*n*-butylsulphonylethyl 1-naphthenate, are useful as plasticisers for polyvinyl chloride and cellulose esters. E. C.

Plasticisers for Vinyl Polymers. Henkel & Cie. *BP* 666,735

Hydroxyalkylamides and dihydroxyalkylamides of subst. or unsubst. benzenesulphonic acids are plasticisers for vinyl polymers. The plasticised polymers are clear and transparent. C. O. C.

Stabilisation of Halogen-containing Macromolecular Substances. N.V. De Bataafsche Petroleum M.-S. *BP* 667,041

Mixtures of a basic-acting substance and an arylurea containing one phenyl group carrying at least one non-hydrocarbon substituent do not separate from halogen-containing polymers to which they are added as stabilisers to heat and light. C. O. C.

Stabilising Polymers or Copolymers of Halogenated Derivatives of Ethylene. Société Anonyme des Manufactures des Glaces et Produits Chimiques de Saint-Gobain, Chauny & Cirey. *BP* 666,970

Alkyd resins obtained by condensing ethylenic dibasic acids with alcohols of general formula $XH_n(CH_2{\cdot}CH_2OH)_m$ (n = 0–2; m = 3–1; X = N or S) are good stabilisers for polymers or copolymers of halogenated derivatives of ethylene. C. O. C.

Colouring Polystyrene. Dow Chemical Co. *BP* 661,761

Aromatic vinyl polymers, e.g. polystyrene, are coloured by mixing in a fluid condition, e.g. in a mixer-extruder at 200°C., with a smaller quantity of a colour concentrate prepared by milling together the polymer and a stable dye or pigment.

BP 662,020

Polystyrene is coloured by polymerising a solution (viscosity > 60 centipoises at 25°C.) in styrene or a mixture of styrene and polystyrene of a colour concentrate prepared by milling together at 180–200°C. a heat-stable pigment and polystyrene. E. C.

Copolymers of Styrene and β-Allyloxyethyl Acrylate. DuP. *BP* 666,725

The copolymers of styrene (40–70) and β-allyloxyethyl acrylate (60–30) are useful as flexible, non-crazing, and inert coating materials. W. G. C.

Accelerator for Amino Resins. British Industrial Plastics. *BP* 662,007

Urea- and melamine-formaldehyde resins are rapidly cured under heat and pressure when 0·05–0·5% on the resin and filler of tetrachlorophthalic acid or anhydride is used as catalyst. E. C.

Ternary Copolymers. Dow Chemical Co. *BP* 661,619

Ternary copolymers suitable for use as foils, coatings, etc. or for spinning into fibres are prepared by copolymerising a mixture of vinylidene chloride (10–60%), α-methylstyrene (10–60%), and acrylonitrile (30–80%). E. C.

Coprecipitated Resins. American Cyanamid Co. *USP* 2,563,898

Aqueous dispersions of water-insoluble thermoplastic resins, e.g. polystyrene or polymethacrylates, can be flocculated or coagulated by mixing with a colloidal cationic melamine–aldehyde resin solution. C. O. C.

Phenol Aldehyde Resins. Lewis Berger & Sons. *BP* 666,557

Alkylphenols (Alk of > 3 C) readily react with acidic formalin at 180–210°F. in presence of a wetting agent containing a sulpho—SO_3M group (M = H, metal, or ammonium) to yield products useful in coating compositions. C. O. C.

Organosilicon Copolymers compatible with Resins, Cellulose Derivatives, Drying Oils, etc. Montclair Research Corpn. and Ellis-Foster Co. *USP* 2,562,953

Copolymers obtained from a silica derivative, e.g. an orthosilicate, and an organosilicon compound are resins of good colour, stability to heat, and chemical resistance. They are compatible with paint-forming materials. C. O. C.

Linear Organosilane Polymers. Dow Corning. *BP* 666,953

Polymers of formula $Y_3Si{\cdot}[C_6H_4{\cdot}SiClY]_n{\cdot}Y$ (Y = Cl or phenyl, at least one Y being phenyl; $n > 0$), useful in coating compositions, are obtained by heating a compound of formula $(C_6H_5)_xSiCl_{4-x}$ (x = 1·5–2·9) in the liquid phase at 210–300°C. in presence of $AlCl_3$. C. O. C.

Polyvinyl Chloride Compositions resistant to Fungi and Bacteria. Monsanto. *USP* 2,567,905

Copper 8-quinolinoxide (0·1–2·5 parts by weight) can be incorporated in polyvinyl chloride compositions (100 polyvinyl chloride), e.g. those used for coating textiles, without blooming or crystallising if there is also added a compound of formula $R{\cdot}SO_2{\cdot}NH{\cdot}Alk$ (Alk of 1–8 C; R = phenyl or tolyl) (5–50).

USP 2,567,910

Up to 10 parts of copper 8-quinolinoxide per 100 of resin can be incorporated if 1–25 parts of the condensate of toluenesulphonamide and formaldehyde is added. C. O. C.

Stable Aqueous Dispersions of Vinyl Ester Neutral Ethylenic Polyester Acrylic Acid Copolymers. Minnesota Mining & Manufacturing Co. *USP* 2,570,253

Film-forming Polyvinyl Acetate Emulsions. Swift & Co. *USP* 2,576,827

The water resistance of films formed from plasticised or unplasticised polyvinyl acetate emulsions is greatly increased if the pH of the emulsion is lowered from the usual 3·5–4·0 to < 2·5. The resulting increase in viscosity can be much reduced by incorporating 0·5–2·5% by weight of quinol into the emulsion. C. O. C.

Hydroxyalkylamine and Morpholine Salts of Polymerised Olefin Sulphonic Acids—Sizes and Coating Compositions (III p. 218).
Stable Aqueous Dispersions of Vinyl Chloride Polymers and Copolymers (III p. 218).
Films containing Metallic Flake Pigments (V p. 224).
Tetramethylurea as Solvent for Polyvinylidene Cyanide (VI p. 227).

XIV—ANALYSIS; TESTING; APPARATUS

Radioactive Tracer Techniques and their Applications in Industry. A. Robson. J.S.D.C., **68**, 7–11 (Jan. 1952).

Determination of Sodium Oleate in Dilute Aqueous Solutions. J. Glazer and T. D. Smith. *Nature*, **169**, 497 (22 March 1952).

A range of solutions of sodium oleate (10^{-4}–10^{-2} M.) are maintained at pH 10 by excess NaOH. Cetyltrimethylammonium bromide (5×10^{-3} M., 10 ml.) is run into a 50-ml. stoppered cylinder containing $CHCl_3$ (10 ml.). The oleate solution is then run in with frequent shaking. Emulsion stability increases as the end-point is approached, and, not before 95% conversion, aqueous Methylene Blue (0·05%, 0·2 ml.) is added as indicator. The true end-point is reached when the dye intensities in aqueous and $CHCl_3$ phases are equal, viewed against diffuse light. As the colour in $CHCl_3$ turns pink after about 1 min. because of the alkali, addition of indicator must not be made until very near the end-point. Results are compared with those from a surface balance method, and excellent agreement is obtained, both methods being accurate to ± 1%. Ca^{++} and Cu^{++} interfere. J. W. B.

Colorimetric Estimation of Dihydric Phenols. D. N. Vaskevich and Ts. A. Goldina. *J. Appl. Chem. U.S.S.R.*, **24**, 1214–1216 (Nov. 1951).

Catechol, resorcinol, and quinol in dil. aq. soln. may be distinguished from one another and estimated colorimetrically by the use of ammoniacal cadmium sulphate as test reagent. A. E. S.

Dye Analysis. II—Detection of Butter Yellow in Fats. H. Thaler and R. Scheler. *Z. Lebensm.-Untersuch. und -Forsch.*, **93**, 286–290 (1951): *Chem. Abs.*, **46**, 1173 (10 Feb. 1952).

Annatto, Chrysoidin R, Martius Yellow, and Sudan G are removed by adsorption on Al_2O_3 and elution with petroleum ether. The ether solution is evaporated to dryness, and the residue taken up with benzene and chromatographed on Floridin XXF. A dark carmine red zone indicates presence of Butter Yellow (*p*-dimethylaminoazobenzene). C. O. C.

Determination of the Solubility of Anionic Leather Dyes. German Society of Leather Technologists. *Das Leder*, **2**, 248 (1951): *J. Soc. Leather Trades Chem.*, **36**, 63 (Feb. 1952).

A Report of the Committee for Leather Dyeing. A proposed standard method is given. C. J. W. H.

Test of the Behaviour of Anionic Dyes in Acid Solution. German Society of Leather Technologists. *Das Leder*, **2**, 248 (1951): *J. Soc. Leather Trades Chem.*, **36**, 63 (Feb. 1952).

A Report of the Committee for Leather Dyeing. A proposed standard method is given. C. J. W. H.

Mechanism of the Gram Reaction. III—Solubilities of Dye Iodine Precipitates and Further Studies of Primary Dye Substitutes. J. W. Bartholomew and T. Mittwer. *Stain Technol.*, **26**, 231–240 (1951).

Provocative Test for Assaying the Dermatitis Hazards of Dyes and Finishes used on Nylon. A. J. Fleming. *J. Invest. Dermatol.*, **10**, 281–291 (1948): *Chem. Abs.*, **46**, 739 (25 Jan. 1952).

Patch tests showed nylon itself to be apparently free from undesirable effects, but of 14 dyes used on nylon some tend to cause skin sensitisation. C. O. C.

X-Ray Diffraction Camera for Fibres and High Polymers. C. Legrand. *J. Polymer Science*, **8**, 337–343 (March 1952).

An X-ray diffraction camera using pure monochromatic light which enables the sample to be inspected in a well controlled atmosphere at a given temperature. The film is kept *in vacuo*. This technique yields diagrams of much improved quality. C. O. C.

Determination of the Degree of Polymerisation of Cellulosic Fibres. H. Vollenbruck. *Melliand Textilber.*, **33**, 153–156 (Feb. 1952).

The reproducibility of D.P. values found for cellulose by the "FaChemFa 2" method (cf. J.S.D.C., **67**, 294 (July 1951)) is not good for natural fibres of high D.P. Satisfactory results are obtained by using a cuprammonium solvent with an increased content of cuprous chloride and of cupric hydroxide, and by wetting out the fibres in conc. NH_4OH before introducing them into the solvent. A. E. S.

Conductometric Titration of the Carboxyl Groups of Cellulose. K. Edelmann. *Faserforsch. und Textiltech.*, **3**, 1–5 (Jan. 1952).

Methods that have been used for estimating the carboxyl content of purified native cellulose are reviewed, and the contradictory results of these methods are discussed. Schmidt's conductometric method (see J.S.D.C., **51**, 152 (April 1935)) is re-examined and found to give reproducible values for a given specimen. The value obtained for cotton cellulose, purified in various ways and electrodialysed before titration, depends on the method of purification. Thus, for raw American cotton it is 0·32% (as CO_2), a value that is reduced only slightly by boiling the cotton in 2% NaOH. After purifying the cotton by ammonium oxalate extraction (removal of pectic substances), no carboxyl can be detected. Even when such purified cellulose is oxidised, e.g. by chlorous acid or nitrogen dioxide, a negative result is obtained. Purified wood celluloses also give negative results. Further experiments show that the method is satisfactory only for low-mol.wt. acids and already shows serious discrepancies for pectic acids. It is of no value for cellulose. A. E. S.

Diazo Compounds in the Determination of Wool Damage. M. V. Glynn. J.S.D.C., **68**, 16–20 (Jan. 1952).

Microkinematics of Textile Processes—Process of Stretching and Breaking Wool (in the Double Loop Test). H. Reumuth. *Melliand Textilber.*, **33**, 41–44 (Jan. 1952).

Two microcinematograph records made in 1942 of the process of fibre extension with rupture have now been evaluated. In the recorded experiments, two wool fibres were looped together; the ends of one loop were clamped at a short distance apart on one of the movable rods of a tensioning device, and the ends of the other loop were clamped in a similar way on the other movable rod. Tensioning was applied by hand, while the double loop was maintained, under glycerol, in the focus of the microscope attached to the ciné camera. Two different tensioning devices were tried; a modified micromanipulator, used in the second experiment, was very satisfactory. In the first experiment, rupture was preceded by tearing and slipping of the epidermis; in the second, an additional feature was the splitting of the fibre in the direction of the fibre axis. Individual pictures, covering ~ 1 s. preceding rupture, are given for each experiment. A. E. S.

Identification of Wool Damage during Singeing. N. van Kuyeren. *Textil-Praxis*, **7**, 41–42 (Jan. 1952).

Damage to wool caused by singeing can be recognised microscopically by the presence of bubbles on the fibre. The size of bubble is proportional to the temp. during singeing. B. K.

Fat Estimation in Textiles. H. Meyer. *Faserforsch. und Textiltech.*, **3**, 45–58 (Feb. 1952).

Recent work on the estimation of fats in textiles is

reviewed, and an account is given of the author's work in search of a solvent of general utility for the fat extraction of all fibres by the Soxhlet method. Such a solvent should not dissolve or attack the fibre, soaps, synthetic detergents, or non-fatty matter. Properties, e.g. water-miscibility, fat-solvent properties, effect on cellulose acetate, inflammability, stability, of 14 common extraction solvents are tabulated. None of these solvents is completely satisfactory, though diethyl ether is rejected only on account of its inflammability and instability. Methylene dichloride, which is non-inflammable and stable, is a good fat solvent and is inert towards soaps and other detergents, but it dissolves or attacks resins, cellulose acetate, and PeCe fibre and other synthetic polymers. A mixture (1 : 1) of diethyl ether and methylene dichloride is found to be a very satisfactory extraction medium and is recommended for general application. It behaves like pure ether towards cellulose acetate and PeCe fibre, and is of low inflammability. Many examples are given of results obtained with this solvent mixture on wool, on cotton (raw, and treated in various ways), and on a wool staple-rayon yarn, treated before or after dyeing with a cation-active agent. A. E. S.

Skin-irritation Tests for the Textile Industry. L. C. Barail. *Amer. Dyestuff Rep.*, **41**, P 136–P 138, P 143 (3 March 1952).

Methods used for testing fabrics, dyes, finishes, and other chemicals in the textile industry for the presence of primary skin irritants and cutaneous sensitisers are discussed, and are compared for accuracy, reproducibility, speed, and cost. Of the patch test on human subjects, the patch test on animals, hypodermic injection into animals, intradermal injection into man, and intradermal single injection into animals, the last is found to be the best screening method. It is simple, cheap, and accurate, and can be used to detect skin irritants and cutaneous sensitisers in 24 hr. The method is described in detail, and it is suggested that negative results should be confirmed by patch tests on human subjects. J. W. B.

Constant Temperature and Humidity Room. J. G. Martindale and J. Angus. *J. Textile Inst.*, **43**, P 122–P 123 (Feb. 1952).

A conditioning and testing room requiring the minimum of maintenance is described. Moisture-permeable and -impermeable areas of the walls are adjusted in such ratio as to permit enough moisture to escape for a "dry tower" to be dispensed with, but not enough to prevent the "wet tower" from being able to top up the humidity. An automatic cut-off prevents flooding should the water waste-pipe freeze up in cold weather. J. W. B.

Measuring Humidity and Testing Hygrometers. A. Wexler and W. G. Brombacher. *U.S. National Bureau of Standards Circular* 512 (28 Sept. 1951).

A review of methods for measuring the humidity of air and other gases and for producing and controlling atmospheres of known humidity for hygrometer testing and calibration and general research. Among hygrometric techniques discussed are psychrometry, mechanical hygrometry, dewpoint measurement, electric hygrometry, gravimetric hygrometry, thermal conductivity, index of refraction, pressure and volume measurements, and dielectric constants. Equipment for establishing desired humidities over a wide range of temperatures is described. 157 references. C. O. C.

Molecular Weight and Viscosity of Polyamides (Perlon, Nylon). F. Leepelmann. *Textilforsch. und Textiltech.*, **3**, 58–66 (Feb. 1952).

In the Kuhn modification of the Staudinger equation, viz. $[\eta] = KN^{\alpha}$, where N is the chain length of a linear polymer (in No. of atoms), the intrinsic viscosity $[\eta]$ may be defined as $\lim_{c \to 0}(\eta_r - 1)/c$, where η_r is the viscosity of a soln. of the polymer relative to that of the solvent, and c is the concn. In practice, extrapolation of $(\eta_r - 1)/c$ is difficult, for at low concn. errors in measurement are great. Extrapolation is aided by using other functions of η_r and c that tend also to $[\eta]$ as a limit. Thus, the function $6(\sqrt[6]{\eta_r} - 1)/c$ has been proposed. It is now pointed out that the above two functions are special cases of $m(\sqrt[m]{\eta_r} - 1)/c$, and it is found, by plotting these functions against c for various values of m, that for solutions of polyamides in m-cresol the value $m = 3$ gives a straight line parallel to the c-axis and permits very accurate extrapolation. Using this procedure, $[\eta]$ is determined for various polycaprolactam preparations, and at the same time N is determined by conductometric end-group titration, as follows— The preparation is shredded and extracted with water to remove monomer and low-mol.wt. fractions. It is dissolved at suitable concn. in m-cresol, and the soln. is diluted with n-propanol and may then be titrated conductometrically with alcoholic NaOH (COOH groups) or alcoholic HCl (NH_2 groups). It is preferred, however, to titrate with one of these and then back-titrate with the other, when more accurate end-points are obtained, giving the total end-groups (COOH + NH_2) to $\sim 1\%$ accuracy. N is found to be related to $[\eta]$ (for $N > 120$) by an equation of the Kuhn form—

$$[\eta] = 3{\cdot}475 \times 10^{-4} N^{0{\cdot}6817}$$

The results are compared with those of other workers. It is generally found that values of N based on COOH content are higher than those based on NH_2 content, and this is considered to be evidence for the occurrence of chain branching with amidine formation. Preliminary results on the osmotic method are given. The method can be applied to polycaprolactam in m-cresol soln., but it is necessary to work at $> 40°$c., or otherwise the viscosity is too high. The value obtained for the mol. wt. of the single preparation examined is in good agreement with that obtained by the end-group method. A. E. S.

Methods for Determining the Diffusion Rates of Macromolecular Substances. K. V. Chmutov and I. Ya. Slonim. *Uspekhi Khimii*, **19**, 142–156 (March–April 1950): *Faserforsch. und Textiltech.*, **3**, 75–77 (Feb. 1952).

A review is made of the methods available for the measurement of the diffusion coefficients of macromolecular substances. There are 20 diagrams and 52 references. Various methods involving the determination of changes in optical absorption or refractive index, as revealed by refraction or interference measurements, are described; these include Chmutov's microcolorimeter, in its application to the diffusion of dyes. The methods of forming the diffusion boundary are discussed. In the cell designed by Chmutov and Slonim, plane stainless-steel surfaces are in sliding contact, and a groove in the upper surface, containing solvent, can be slid into position over a groove in the lower surface, containing soln. The ends of the grooves are sealed by contact with the optical flats that close the cell at each end.

The long abstract in German of the above Russian paper contains 7 diagrams and 36 references. A. E. S.

Provisional Standard Methods for Assessing the Colour Fastness of Textiles. French, German, and Swiss Fastness Committees. *Melliand Textilber.*, **33**, 72–83 (Jan. 1952); *Textil-Praxis*, **7**, 157–160 (Feb.), 217–219 (March), 305–308 (April), and 373–374 (May 1952); *Textil-Rund.*, **7**, 27–29 (Jan.) and 71–73 (Feb. 1952).

Comment on the Standard Methods. P. Rabe. *Melliand Textilber.*, **33**, 70–72 (Jan. 1952).

A comprehensive range of proposed standard specifications for the assessment of the fastness of dyeings is given. These are based on the conjoint work of the French, German, and Swiss Fastness Committees and were accepted by them and, conditionally, by delegates of organisations of ten other European countries at a meeting at Basle in February 1951. They are prefaced by a discussion on fastness testing. A short survey is given of the history and work of the various national organisations concerned with fastness testing, and the former standard methods of the Deutsche Echtheitskommission are compared with the present proposals. The new methods are distinguished by the general use of fixed standard depths in various hues for establishing the depths at which testing should be done and by the use, except when testing for light fastness, of fixed standard ranges of five grey depths as an aid to judging degree of staining or loss in depth; the latter obviate the use of the multiplicity of type dyeings previously employed. The idea of using a simple fixed grey scale was developed both on the Continent and in England from more complicated systems employing scales in various hues; there was also a 24-degree grey disc method, which allowed comparisons to be made for

any depth of dyeing and was at one time approved by the Swiss and French. The hope is expressed that the issue of the specifications will mark a step towards complete international agreement on fastness testing. A. E. S.

A Munsell Colour Book in Textiles. H. R. Davidson and H. Luttringhaus. *J. Opt. Soc. Amer.*, **41**, 623–625 (1951): *J. Textile Inst.*, **43**, A 104 (Feb. 1952).

A colour atlas based on the Munsell notation as revised by the Optical Society of America has been prepared using dyed wool flannel. This has the advantage for textile users that colour comparison is made easier, since there is less difference in texture than when using paper samples. The range of colours available is different from that in the Standard Munsell Book of Colour on account of the different colouring matters used and the different surface structure. The dyes used were selected for their light fastness and brightness. The arrangement was made on the basis of physical colorimetry using the Hardy recording spectrophotometer and an automatic integrator. The atlas has been constructed primarily for the authors' own use. C. J. W. H.

Colour Measurement. H. J. Selling; E. I. Stearns. *Amer. Dyestuff Rep.*, **41**, P 71–P 73 (4 Feb. 1952).

Comments on the paper on spectrophotometry by Stearns (*ibid.*, **40**, P 563 (3 Sept. 1951)) and a brief survey of the photoelectric colorimeter method of colour matching employed by the Vezelinstituut TNO, Delft, Holland. Reply by Stearns. J. W. B.

Rates of Fading of the S.D.C. Light Fastness Standards (*B.S.*1006). R. H. Ricketts. J.S.D.C., **68**, 200–203 (June 1952).

Fading Lamps. P. Rabe. *Textil-Praxis*, **7**, 148–150 (Feb. 1952).

Fading lamps employing a carbon arc give the most rapid results and are perfectly reliable for comparing routine dyeings prepared with similar products. Care must be taken in evaluating the results of exposures of different dyes and unknown combinations. Good results are obtained with low-voltage lamps and a suitable filter such as the apparatus designed by Rein. A preliminary examination with a 2000-watt Osram-Nitra lamp gives a good general indication of fastness. Ultraviolet lamps are quite unsatisfactory. B. K.

"Heliotest"—A New Rapid Light-fastness Tester. F. Gasser and H. Zukriegel. *Melliand Textilber.*, **33**, 44–46 (Jan. 1952).

In the light-fastness tester described, light from the direction of the sun is collected by lenses and concentrated on the patterns under test, so as to give a 25-fold increase in intensity. The system is oriented automatically by means of a heliostatic drive. The results are claimed to be highly consistent with those obtained by the normal method of exposure. A. E. S.

Preparation of Equivalent Dyeings and their Application to the Assessment of Artificial Daylight. G. Thomas. *Melliand Textilber.*, **32**, 936–938 (Dec. 1951).

The behaviour of a number of dyes in daylight and artificial light, together with emission curves, is discussed. Dyeings which, although possessing different absorption curves, appear identical in daylight will in general differ in appearance under other types of illumination, and may thus be employed for assessing daylight lamps. Lists of such "conditionally equivalent" pairs of dyes are given. Some details are given of a fluorescent daylight lamp "HNT", which is stated to give good results in practice. B. K.

Effects of Fabric Construction and Mercerisation on the Light and Wash Fastness of Vat Dyes. A.A.T.C.C. Southeastern Section. *Amer. Dyestuff Rep.*, **41**, P 173–P 176 (17 March 1952).

Mercerised and unmercerised cotton fabrics are dyed with selected vat dyes. Penetration is estimated from microscopic examination of warp and filling cross-sections, and light and wash fastnesses are assessed by standard methods. Similar variations in penetration are found with both types of fabric, but the depth of shade of the mercerised cotton is distinctly greater. Light and wash fastnesses are characteristic of the dye, and are unaffected by either fabric construction or previous mercerisation. J. W. B.

Methods of Determining Iodine Absorption, Reduced Iodine Absorption, and Degree of Mercerisation. K. Schwertassek. *Faserforsch. Textiltech.*, **3**, 87–95 (March 1952).

Experimental details are given of the author's diagnostic methods for examining the effects of various processes and degradation treatments on cellulosic materials. The methods depend on the determination of the iodine absorption of the material itself, of the material after a standard steaming treatment, and of the material after a standard mercerisation treatment. A. E. S.

Detection of Synthetic Resins in Crease-resisted Viscose Rayon with Tollens' Reagent and with Kiton Pure Blue V. M. Kramer and W. Graeser. *Melliand Textilber.*, **33**, 226–229 (March 1952).

A critical examination is made of Bernegger's method (see J.S.D.C., **65**, 323 (June 1949)) of staining microscopic sections of crease-resistant textiles with Tollens' reagent. It is found that the method gives poor reproducibility, and some of Bernegger's observations on finishes obtained in a specified manner cannot be repeated. Difficulties arise also when degraded cellulose is present, for this is stained under the conditions of the test. It is concluded that the reaction does not proceed topochemically. On the other hand, it is confirmed that staining with a soln. of Kiton Pure Blue V yields good, consistent results, giving a clear indication of the distribution of the resin across the section. This stain can be applied also to fabric, and gives some indication by its hue of the condensation conditions that have been used. A. E. S.

Determination of the Resistance to Beetles of Wool and other Keratinous Materials. Schweizerische Normen-Vereinigung, SNV 95 902. *Textil-Rund.*, **7**, 115–117 (March 1952).

Notes on the Standard. O. Wälchli and R. Zinkernagel. *Ibid.*, 105–113.

The tests specify the employment of the carpet beetle, *Anthrenus vorax* W. Full experimental details are given and the method is critically discussed. G. L.

Quantitative Estimation of Eulan CN, Mitin FF, and Lanoc CN in Wool. R. S. Hartley, F. F. Elsworth, P. G. Midgley, and J. Barritt. J.S.D.C., **68**, 171–175 (May 1952).

Determination of Copper in Pulp and Paper—Investigation of a New Reagent for Copper. C.-U. Wetlesen and G. Gran. *Svensk Papperstidning*, **55**, 212–216 (31 March 1952).

A method is described based on the blue colour given by the very sensitive reaction between copper in weakly alkaline solution and bis*cyclo*hexanone oxalyldihydrazide. S. V. S.

Ethoxy Determination in Ethyl Cellulose. C. A. Redfarn and D. R. Newton. *Chem. and Ind.*, 404 (3 May 1952).

In a determination of ethoxy content of ethyl cellulose, using a Zeisel-type apparatus, erratic values of methoxy content are obtained with a control sample of pure vanillin. It is suggested that *B.S.* 572 : 1934 ground glass joints are not adequately gas-tight, as good results are obtained with old-fashioned apparatus. J. W. B.

Determination of Alkoxyl Groups. I—Description of a Modified Apparatus. G. Gran. *Svensk Papperstidning*, **55**, 255–257 (15 April 1952).

A modified apparatus suitable for serial analysis is described, in which two washing tubes are provided to enable higher gas velocity than usual to be applied and which can contain different liquids if desired. S. V. S.

Determination of Alkoxyl Groups. II—Determination of Alkoxyl Groups in the presence of Sulphur-containing Substances. G. Gran. *Svensk Papperstidning*, **55**, 287–290 (30 April 1952).

About 50 mg. of the test substance and a known amount of sodium sulphate are weighed accurately into the reaction flask of a modified methoxyl apparatus fitted with two washing tubes, and a few mg. of red phosphorus and 5 ml. of hydriodic acid added. The flask is heated in an oil bath at ca. 140° C. and a stream of carbon dioxide or nitrogen passed through the apparatus. After leaving the condenser the gas stream passes through the two washers and then into the receiver containing 10 ml. of absorption solution (sodium acetate in glacial acetic acid, 150 g./litre) and 0·5 ml. of bromine. After 1½–2 hr. the contents of the

receiver are emptied into a 500-ml. wide-mouthed Erlenmeyer flask, and 15 ml. of sodium acetate solution added. Excess bromine is removed by adding a few drops of formic acid, 1 g. of solid KI and 20 ml. of dil. H_2SO_4 are added, and the resulting solution is titrated with 0·1 N. sodium thiosulphate. A blank is run in exactly the same way. The first washing tube contains 2–3 ml. of distilled water to trap any HI, and the second 5–10 ml. of 5% cadmium sulphate solution to precipitate H_2S. The gas rate should be about 40 ml./min. S. V. S.

Detection of Damaged, Bleached, or Dyed Human Hair. B. Mueller and H. Barth. *Deutsch. Z. ges. gericht. Med.*, **40**, 553–560 (1951): *Chem. Abs.*, **46**, 2758 (25 March 1952).

Mechanically damaged or peroxide-bleached hair turns red when treated with diazotised sulphanilic acid in 5% Na_2CO_3. Dyed hair dissolved in 10% NaOH often lightens on addition of 30% formaldehyde. C. O. C.

New Method for Peptide Degradation. F. Wessely, K. Schlögl, and G. Korger. *Nature*, **169**, 708–709 (26 April 1952).

A scheme for identifying peptide terminal groups is presented, using tripeptides as examples. Conversion to a carbalkoxy compound is followed by heating with 2 mol. of alkali to give a urea derivative. HCl treatment gives a simple amino acid and a hydantoin, which is easily separated, hydrolysed, and identified by paper chromatography. Clear results are obtained with known tripeptides. J. W. B.

Determination of the Particle Size of Dispersed Resins by Two Optical Methods. E. Atherton and R. H. Peters. *J. Textile Inst.*, **43**, T 179–T 187 (April 1952).

Measurements of turbidity and dissymmetry are made on samples of commercial Cellosolve methacrylate resin using a spectrophotometer and a differential refractometer. Results agree well, but are smaller than those obtained with the electron microscope; the discrepancy is due to particle aggregation, and can be overcome by ignoring those particles on the micrograph which, on close examination, appear to be of a compound nature. J. W. B.

Photoelectric Turbidimeter for Particle Size Determination. E. Atherton. *J. Textile Inst.*, **43**, T 173–T 178 (April 1952).

A turbidimeter has been designed for measuring the intensity of light scattered from a suspension or solution contained in an optical cell, over a range of angles from 40° to 150°. It is useful for investigating particle size of textile-bonding resins. J. W. B.

Determination of Rubber Hydrocarbon by Modified Bromination Method. W. J. Gowans and F. E. Clark. *Anal. Chem.*, **24**, 529–533 (March 1952).

Infrared Determination of Free Phenol in Phenol Formaldehyde Resins. J. J. Smith, F. M. Rugg, and H. M. Bowman. *Anal. Chem.*, **24**, 497–498 (March 1952).

The method is based on the strong infrared absorption of phenol at 14·4 μ. An acetone soln. of the resin is used for the determination, and as little as 0·2% unreacted phenol may be detected. Advantages are that procedure is rapid, the absorption max. at 14·4 μ. is not shown by combined phenol, and no preliminary separation of the phenol is needed. J. W. D.

Polarographic Determination of Acrylonitrile. W. L. Bird and C. H. Hale. *Anal. Chem.*, **24**, 586–587 (March 1952).

Polarographic Determination of Methacrylonitrile. L. J. Spillane. *Anal. Chem.*, **24** 587–588 (March 1952).

Determination of Methyl Methacrylate by Oxidative Titration. S. dal Nogare, L. R. Perkins, and A. H. Hale. *Anal. Chem.*, **24**, 512–515 (March 1952).

PATENTS

Detergent-testing Machine. R. Bernstein. *USP* 2,568,707

A paddle wheel, the paddles of which are frames for holding specimens of cloth, revolves in a tank, so that the cloth is repeatedly treated by the detergent liquor. C. O. C.

Moisture-measuring and -controlling Instruments. Standard Telephones & Cables. *BP* 668,196

A humidity-sensitive element consists of glass fibres loosely packed between two plate electrodes and in electrical contact with them. The resistance between the electrodes thus varies with the amount of moisture on the fibres in equilibrium with the moisture content of the surrounding atmosphere. C. O. C.

Photoelectric Colorimeters. A. L. M. A. Rouy. *BP* 666,886

A photoelectric colorimeter comprises a light source, filter, container for the light-absorbing medium, a photoelectric cell, and a microammeter; the cell may be rotated by exterior mechanical means so that it is no longer at right-angles to the light beam. When testing a solution with a minimum light absorption, e.g. a salt solution, which is in effect one of zero dye concentration, the microammeter reading may well be off the scale. By rotating the cell the photoelectric effect is reduced and the reading brought back to the end of the scale, thus adjusting the colorimeter to the best advantage for that particular range of concentrations. J. W. B.

Colour Comparator. A. J. Bruning. *USP* 2,566,079

A colour-measuring and -matching device which casts the image of the sample to be measured or matched on to a screen, another image being formed alongside it through standard colour members. C. O. C.

Measuring and Registering Fibre Lengths. Wool Industries Research Assocn. *BP* 666,492

Measuring the Brightness of White Substances. D. W. Kent-Jones and A. J. Amos. *BP* 669,950

Apparatus for measuring the whiteness of flour or starch. C. O. C.

Colour and Colour Difference Meter. Henry A. Gardner Laboratory. *USP* 2,574,264

A photoelectric tristimulus colorimeter for directly measuring three numerical values for identifying a colour by the co-ordinates of a colour solid having scales of approximately uniform colour-perception spacing. C. O. C.

Colorimeter. R. Leiser. *BP* 670,780

The colour required to match the sample is blended additively by colour filters placed over a diaphragm aperture between a source of light and a lens system for providing a focussed real image of the light source. The colour filters are mounted in a rectangular frame on one side of a plane passing perpendicularly through two opposite side-members of the frame. On the other side of the plane there is in the frame means for transmitting white light. The frame can be displaced in a plane parallel to the plane of the diaphragm aperture and in either of two mutually perpendicular directions. This makes full use of all the light and enables reproducible results to be readily obtained by different observers. C. O. C.

Static Problems and their Control in the Textile Industry (VI p. 224).

[Spectroscopic Estimation of Metals in Wool] (VI p. 226)

Textile Application of Brighteners (VII p. 227).

XV—MISCELLANEOUS

PATENTS

Harmless Hair-dyeing Creams. Union Française Commerciale & Industrielle. *BP* 671,122

A hair-dyeing cream comprises a carrying cream, a hair dye which requires no chemical treatment, e.g. oxidation, and a wetting agent. C. O. C.

Coloured Crayon or Pencil. National Cash Register Co. *BP* 670,867

An erodable base material, e.g. gelatin, has dispersed in it an adsorbate compound, e.g. Crystal Violet lactone, so that when it is drawn across a surface sensitised with an adsorbent material, e.g. kaolin, a coloured mark is produced. C. O. C.

Condensation Products from Waste Superpolyamide Fibres. E. E. Verdiers. *BP* 670,693

Waste nylon or the like is heated with formic, sulphuric, or hydrochloric acid or other degradation agent so as to destroy its filamentary nature, and is then precipitated from the mixture and ground into powder. The product, especially if it is mixed with silk fibroin, is used in soap and cosmetics; e.g. when dyed it forms a translucent face powder. C. O. C.


crocking
problem
mastered
Aridye
pigment colours

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MANAGER required for bleaching, dyeing, printing and finishing mill in Lebanon, handling cotton and viscose yarn and piece goods. Essential applicants be capable of supervising entire production and taking complete control of bleaching, dyeing and finishing section. Plant consists of conventional type yarn and piece goods dyeing and finishing machinery including du Pont continuous bleacher, pad steam dyeing unit, package dyeing unit, Sanforised shrunk controlled compressive shrinkage unit, using Vat, Azoic and Sulphur dyes. Individual with overseas experience and knowledge of French or Arabic preferred. Attractive terms and conditions. Details at interview. Box V572

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MISCELLANEOUS

THE proprietors of British Patent No. 666896 for "Improvements in or relating to the Dyeing of Artificial Fibres" are prepared to enter into negotiations for the SALE of the patent or for the grant of a LICENCE thereunder. Inquiries to be addressed to Carpmaels & Ransford, 24 Southampton Buildings, Chancery Lane, London W.C.2.

WANTED—Copy of bound volume on Symposium on Fibrous Proteins. Box M448

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Manuscripts submitted for publication in the *Journal* should be typewritten (double spacing) on good-quality paper, using one side of the paper only and leaving a margin at least 1 in. wide on the left-hand side. The time taken in refereeing papers (both lectures and communications) will be reduced to a minimum when authors submit two copies of the typescript.

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The list of references should be given at the end of the manuscript and the abbreviations used should be, as far as possible, those given in the "List of Periodicals Abstracted" included at the end of the index to the preceding year's *Journal*. Reference numbers in this list should be neither enclosed in brackets nor followed by full-stops. As far as possible throughout the manuscript the abbreviations listed in the Jan. 1950 issue (p. 54) should be used. Tables should be numbered consecutively in Roman numerals and Figures in Arabic numerals.

The number of figures and graphs should be kept as low as possible, and data should be presented in the form of either tables or graphs, not both. Drawings should be carefully prepared, preferably in Indian ink, on plain white drawing paper or, preferably, Bristol board. In graphs, the frame and actual curves should be ruled and inked more heavily than any co-ordinate lines, and the latter should not be close together as in ordinary graph paper. Experimental points should always be given, and where several graphs appear in a single Figure clear means of differentiation must be adopted. All numbers and legends are set up in type by the printer, and authors should therefore indicate them lightly in pencil.

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